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CORROSION INHIBITING ENGINE OILS

Pratt & Whitney Aircraft Group
Government Products Division
West Palm Beach, Florida 33402

August 1981

Interim Report for Period 4 September 1979 - 1 December 1980

Approved for Public Release; Distribution Unlimited

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AIR FORCE WRIGHT AERONAUTICAL LABORATORIES
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This technical report has been reviewed and is approved for publication.

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The Fluids, Lubricants, and Elastomers Branch of the Materials Laboratory, Air Force ~~Wright Aeronautical Laboratories~~ established the requirements for a MIL-L-7808H operational oil with corrosion inhibition characteristics at least equal to those of MIL-C-8188C. The new oil formulation is to provide corrosion inhibition under long-term storage conditions of the ALCM F107 turbine engine. During the initial fifteen-month effort, several approaches were evaluated as candidate methods for determining the effectiveness of corrosion inhibiting oil

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FOREWORD

This report describes the technical effort conducted by the Advanced Fuels and Lubricants Group of United Technologies Corporation, Pratt & Whitney Aircraft Group, Government Products Division under Contract F33615-79-C-5089, Project ILIR, Task 01, Work Unit 25, Corrosion Inhibiting Engine Oils. This program is under the direction of Dr. Wayne E. Ward of the Fluids, Lubricants, and Elastomers Branch of the Materials Laboratory, Air Force Wright Aeronautical Laboratories, Wright-Patterson Air Force Base, Ohio.

The technical effort disclosed herein was performed during the period 4 September 1979 to 1 December 1980 under the direction of the Pratt & Whitney Aircraft Group Program Manager, Mr. Grayson C. Brown. The report was released by the authors in March 1981.

This report is an interim Report concerned with the development and laboratory analysis of corrosion inhibiting additives for use with current MIL-L-7808H type lubricating oils. The final formulation is to provide corrosion protection under long-term storage conditions and be an operational lubricant in the Williams Research Company F107 turbine engine. A Final Report will follow at the conclusion of the technical effort.

ACKNOWLEDGEMENTS

The candidate corrosion inhibitors evaluated in this program were acquired through the cooperation of the manufacturers listed below:

Bray Oil Company
9550 Flair Drive
Suite 301
El Monte CA 91731

Edwin Cooper Incorporated
Subsidiary of Ethyl Corp.
Route 3
Sauget IL 62201

E. I. DuPont & Co., Inc.
Petroleum Chemicals Division
Wilmington DE 19898

Minnesota Mining & Mfg. Co.
3M Center
St. Paul MN 55101

Mooney Chemicals Incorporated
2301 Scranton Road
Cleveland OH 44113

Nalco Chemical Company
Petroleum Chemicals Division
Sugarland TX 77478

Pfaltz & Bauer Incorporated
375 Fairfield Avenue
Stamford CT 06902

Ronco Laboratories Incorporated
3617 Brownsville Road
Pittsburg PA 15227

The Lubrizol Corporation
19400 Lakeland Blvd
Wickliffe OH 44092

Universal Oil Products Company
UOP Process Division
Des Plaines IL 60016

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LIST OF ABBREVIATIONS

AFWAL	Air Force Wright Aeronautical Laboratories
AI	Active ingredient
ALCM	Air-launched cruise missile
AQL	Acceptable Quality Level
atm	Atmospheres
ASTM	American Society of Testing and Materials
cc/min	Cubic centimeters per minute
CCI	Candidate corrosion inhibitors
cm	Centimeters
COS	Corrosion Oxidation Stability
CR	Corrosion rate
CREP	Corrosion Rate Evaluation Procedure
cSt	Centistokes
DI	Deionized (water)
dia	Diameter
FTM	Federal Test Method
g	Grams
hr	Hours
in.	Inches
mg	Milligrams
min	Minutes
ml	Milliliters
MLBT	Fluids, Lubricants and Elastomers Branch, Materials Laboratory, AFWAL
mm	Millimeters
MN/m^2	Meganewtons per square meter
N	Number of corrosion rate determinations
NBS	National Bureau of Standards
pH	Hydrogen ion concentration
PMC	Product Material Control
P&WA/GPD	Pratt & Whitney Aircraft, Government Products Division
QPL	Qualified Products List
QRN	Quality Reference Number
rcf	Relative centrifugal force
RQL	Rejectable Quality Level
sec	Seconds
SEM	Scanning electron microscopy
SSP	Sequential Sampling Plan
₹	Standard taper
TAN	Total acid number
UTC	United Technologies Corporation
v/o	Volume percent
w/o	Weight percent
WPAFB	Wright-Patterson Air Force Base

SUMMARY

The Fluids, Lubricants, and Elastomers Branch of the Materials Laboratory, Air Force Wright Aeronautical Laboratories established the requirements for a MIL-L-7808H operational oil with corrosion inhibition characteristics at least equal to those of MIL-C-8188C. The new oil formulation is to provide corrosion inhibition under long-term storage conditions of the ALCM F107 turbine engine.

During the initial fifteen-month effort, several approaches were evaluated as candidate methods for determining the effectiveness of corrosion inhibiting oil additives. A screening procedure was developed after preliminary modifications were made to the Corrosion Rate Evaluation Procedure (CREP). Using the CREP together with compatibility and miscibility screening of oil/inhibitor blends, a total of 67 candidate corrosion inhibitors (CCI) were evaluated. These included CCI obtained from Bray Oil Company, El Monte, California, as part of a subcontract to synthesize candidate corrosion inhibitors. Additional tests were completed on a preliminary physical and chemical property evaluation of the oil/inhibitor blends in regard to MIL-L-7808H and MIL-C-8188C specifications.

At the conclusion of this work, it was determined that an improved CREP should be developed for meaningful comparison to the Humidity Cabinet Corrosion Test in ASTM D-1748 as part of a redirection of effort before extensive property evaluation can be concluded.

SECTION I

INTRODUCTION

Engine lubricants must provide corrosion protection to static engine parts, such as gears, bearings, and other moving parts, during interim periods between engine operations. In the case of the Air Force Air-Launched Cruise Missile (ALCM), a storage time of 30 months is required. This is a static, non-controlled storage with no exercising of the engine to maintain or re-establish a protective oil film. A previously developed corrosion-inhibiting version of MIL-L-7808H type engine oil, specified as MIL-C-8188C, has been found to be limited in performance characteristics and long-term corrosion inhibiting protection.

This program directly addresses the development and testing of oil/inhibitor blends that will provide the necessary protection and operational characteristics to permit uncompromised operation of the F107 engine. The program was initially divided into four technical tasks, as noted below:

Task 4.1	Literature Search and Custom Synthesis
Task 4.2	Test Procedure Development
Task 4.3	Formulation and Evaluation of Oil/Inhibitor Blends
Task 4.4	Elastomeric and Corrosion Oxidation Stability (COS) Evaluations

Each of these tasks was proposed as a logical sequence leading to the development of an improved corrosion inhibiting MIL-L-7808H type oil.

Task 4.1 was divided into two subtasks, the first of which involved the search of current technical literature on corrosion and corrosion inhibition. This search, together with key words and reference sources, is discussed in detail in Section II of this report. In addition to the literature search, this task initiated communications with potential manufacturers of possible candidate corrosion inhibitors (CCI). Samples from each manufacturer were requested based on P&WA/GPD requirements, as outlined in P&WA/GPD proposal FP79-156 dated 24 April 1979.

Also initiated and completed under Task 4.1 were the custom syntheses of CCI. These syntheses were conducted by the Bray Oil Company of El Monte, California. Initially, a four-month time period was given for the completion of both subtasks. The literature search was completed on time, but a four and one-half month extension was given to Bray Oil Company by the GPD Program Manager in order to finalize the chemical development of CCI.

Task 4.2 addressed the development and checkout of an analytical test method for the candidate corrosion inhibitor evaluations. Various techniques were used in order to comparatively evaluate the corrosion protection provided by MIL-L-7808H and MIL-C-8188C lubricants.

After these methods were tested, a single Corrosion Rate Evaluation Procedure (CREP) was developed, improved, and later verified as a screening test to be used for evaluation of each CCI.

The technical effort of Task 4.3 involved the screening and evaluation of 67 CCI on the basis of their solubility characteristics in MIL-L-7808H oil, and their corrosion protection capabilities as determined with the CREP developed in Task 4.2. The CCI were screened to select the 13 most promising, which were subsequently evaluated at various concentrations in MIL-L-7808H to determine their effect on the physical or chemical properties of the matrix oil.

Work entailed in Task 4.4 was not initiated because of the need to improve the accuracy and precision of the CREP and in turn decrease its severity. Further work to modify the procedure is underway based on the conclusions of this phase of the program.

SECTION II

TASK 4.1 - LITERATURE SEARCH AND CUSTOM SYNTHESIS

A complete literature search was conducted to comply with the requirements of Contract F33615-79-C-5089 to evaluate the state of the art in corrosion inhibitors for MIL-L-7808H type aircraft engine lubricants. The key words selected for use in the literature search were based on the topic of corrosion inhibitors; i.e., corrosion inhibition, corrosion inhibitors, lubricant additives, lubricating oils. Several sources of information were utilized to perform the literature search, including a thorough search of DIALOG™ (Lockheed Information Systems), Corrosion Abstracts/Florida Atlantic University, and the Defense Documentation Center of the Defense Logistics Agency. The literature search included review of over 8,000,000 citations. A list of articles available was scrutinized to select only those references related to the program. The articles were ordered through the P&WA/GPD Library Branch of the United Technologies Corporation Library. A copy of all references was indexed and sent to the AFWAL/MLBT Program Manager on 28 March 1980. The literature search is being updated continuously as new articles are received. A summary of the reference sources is given below.

1. DIALOG™ (Lockheed Information Systems, Palo Alto, California)

   a. Comprehensive Dissertation Abstracts (CDA) includes university dissertations of over 630,000 citations with monthly updates from virtually every American college and university within the time span of 1861 to December 1980.

   b. Claims™ Patents was searched using five definitive data bases, as listed below:

      - "Claims/Chemistry: From 1950 through 1970" included over 265,000 sources of US chemical and chemically related patents issued during this time period. These also included foreign equivalents from Belgium, France, Great Britain, West Germany, and the Netherlands.

      - "Claims/Class" is the classification code and title directory for all classes and selected subclasses of the US Patent Classification System. Over 15,000 sources were reviewed using this data base in order to facilitate the other searches.

      - "Claims/US Patents: From 1971 to 1978" which contains over 485,000 records utilizing quarterly updates of all US patents was reviewed for corrosion inhibiting additives in oil lubricant systems.

      - "Claims/US Patents: From 1978 to the Present" contains over 85,000 records reviewed as above for corrosion inhibitors for engine oil systems.

- "Claims/US Patent Abstracts Weekly" was reviewed in order to supplement the above two Claims for the month of October 1979 only. A total of 3000 citations were reviewed.

c. Metadex (Metals Abstracts/Alloys Index) from 1966 to present contains over 370,000 citations with monthly updates from the American Society of Metals. These citations were reviewed for corrosion inhibitors.

d. Scisearch^{R}, a multi-disciplinary index to the literature of science and technology, was reviewed to the full extent of its 2,700,000 citations. References searched were derived from over 2600 of the major scientific and technical journals.

e. ISMEC, the Information Service of Mechanical Engineering indices, provided a review of the significant articles of mechanical engineering from approximately 250 journals published throughout the world. This search included over 90,000 citations for corrosion inhibitors.

f. CA Search, covering Chemical Abstracts from 1967 to the present, including some 4,000,000 citations, was reviewed in its entirety.

g. Smithsonian Science Information Exchange (SSIE) Current Research includes only the last two years, or some 253,000 citations. This data base contains reports of both Government and privately funded scientific research projects from over 1300 organizations that fund research.

h. NTIS, National Technical Information Service, includes citations from reports of Government-funded studies.

i. COMPENDIX, an Engineering Index, reviews magazines and articles in the engineering field.

j. Conference Papers Index reviews national and international conference presentations documented by publications in the area of scientific research and development.

2. Corrosion Abstracts/Florida Atlantic University. These abstracts were reviewed through the reference library of Florida Atlantic University at Boca Raton, Florida. The abstracts cover corrosion and its related effects, as published in technical journals of the National Association of Corrosion Engineers.

3. Defense Documentation Center of the Defense Logistics Agency in Alexandria, Virginia includes reports from Wright-Patterson Air Force Base, Naval Air Development Center, Southwest Research Institute, and other Government agencies.

The Bray Oil Company of El Monte, CA has completed the synthesis of several candidate corrosion inhibitors (CCI) under a subcontract to

P&WA/GPD. These CCI were incorporated into the total number of CCI evaluated in this program. A final report was written by Bray Oil Company, and copies were previously submitted to both the P&WA/GPD and AFWAL/MLBT Program Managers.

SECTION III

TASK 4.2 - TEST PROCEDURE DEVELOPMENT AND VALIDATION

The primary objective of Task 4.2 was the development of an effective, accelerated characterization procedure for the evaluation of the corrosion protective capabilities of candidate formulations.

One method used to quantitatively evaluate the degree of corrosion is based on the use of buffered acidic solutions. The selection of acid buffers was predicated on the acid level found in the precipitation of various locations in Europe and the United States (References 1 and 2). Initially, a mixture of acids (2% hydrochloric acid, 28% nitric acid, and 70% sulfuric acid) that contained no more than 100 parts per million of sodium, potassium, and calcium salts was used to prepare a solution of pH 4.5. One-hundred milliliters (ml) of the solution were added to a series of four 1,000 ml PYREX^{TM} reaction kettles and heated to 100°C (212°F). Sample specimens were fabricated from 1.25 x 5.08 x 0.16 cm (0.5 x 2.0 x 0.06 in.) strips of AISI 1010 low-carbon cold-rolled sheet stock. These strips were drilled with a 0.24 cm (0.09 in.) dia hole at one end for suspension within the reaction vessel. Each strip was prepared to a surface finish of 10 to 20 microinches with 240 grit abrasive paper and cleaned in boiling toluene, then in boiling acetone prior to weighing. Typical weights were approximately 7g. The reaction kettles were fitted with a four-hole ground glass lid. Two of the lids were fitted with Graham-type condensers and the remaining two lids were fitted with Allihn-type condensers. All condensers were water-cooled and all reaction kettles were supplied with Celsius thermometers with bulbs positioned 7.5 cm (2.9 in.) above the buffered solution. Each reaction system was placed on a heating element and supplied with a Teflon encapsulated stirring bar. The acidic solution was initially stirred to ensure an even temperature distribution of the hot vapor within the chamber. A series of tests was performed to compare corrosion on protected (MIL-C-8188C) and unprotected strips. The results indicated that stirring was not necessary if the kettles were heated to 100°C (212°F) and allowed to reach thermal equilibrium prior to introduction of the sample strips. Thirty minutes was found to be adequate time for equilibration. The sample strips were suspended from 21-gauge Inconel wire inside the reaction kettle approximately 5 cm (2 in.) above the boiling solution. These wires were inserted through a No. 5 rubber stopper and suspended in the temperature equilibrated reaction kettle.

A series of 24 tests was conducted to determine the nominal length of time for meaningful test runs. These tests were conducted using a 0.15 molar acetic acid solution of pH 4.5. An effective minimum residence time was found to be 3 hours. No significant corrosive attack was observed on test specimens suspended above the acetic solution. An increase of two pH units occurred during the testing (ie., 4.5 to 6.5).

The next solution to be evaluated was a buffer solution compounded from citric acid and sodium citrate adjusted to a pH of 5.0. Samples from initial testing exhibited a mild degree of corrosion. Four test fluids were subsequently made up of acetic acid solutions (pH 5) combined with up to 0.5 molar concentrations of sodium, potassium, and calcium salts. Tests performed at 100°C for 3 hr, with four acid solutions, showed only very mild corrosive effects. The final solution of this series to be evaluated was a certified buffer solution obtained from Fisher Scientific (sodium acetate, So-B-100). The pH of this solution was 4.63 with a molar concentration of 0.1 total acetate. Preliminary results showed appreciable corrosion after 3 hr at 100°C. Weight loss for cleaned, unprotected strips was approximately 3 to 6 mg using this buffer solution compared to erratic results observed with the other solutions during the 3-hr test.

In order to validate reproducibility, multiple tests were run using identically prepared samples. One-half were coated with MIL-C-8188C oil and the other half were unprotected. Results varied gravimetrically from 0.1 to 10.0 mg on identical tests using split samples from the same aliquot of oil.

A problem was believed to exist in the sample preparation procedure that consisted of cleaning the post-test specimens with acetone prior to weighing. A series of tests was then completed in an effort to establish a procedure for sample strip preparation. The procedure was as follows:

1. A solution of 15 volume percent (v/o) of concentrated hydrochloric acid was homogeneously blended with 1.5 v/o of P&WA Specification PMC 1015 (Inhibitor for Muriatic Acid)

2. Sample strips were prepared by surface finishing with 240-280 grit alundum abrasive paper, then immersed for 10 sec in the hydrochloric acid solution, and immediately immersed in dilute reagent-grade sodium hydroxide (0.1 molar)

3. Samples were then immersed in boiling toluene followed by boiling acetone, flash dried, cooled in a desiccator, and weighed to $\pm$0.1 mg.

Corrosion tests run on unprotected samples using this cleaning procedure prior to testing improved reproducibility compared to samples cleaned by any of the previous methods. As expected, the tests conducted using MIL-C-8188C coated samples as opposed to unprotected samples provided greater protection against corrosive attack.

Simultaneously, a sonic cleaner was under study for coating strips with oil/inhibitor combinations prior to introduction into the corrosive environment. This approach proved time-consuming with no apparent advantage gained over coating by immersing the sample strips in a beaker containing the oil/inhibitor combination. Work was also ini-

tiated at this time using a Corrosion Oxidation Stability (COS) apparatus manufactured by Roxana Machine Works, South Roxana, Illinois. The unit can test up to eight specimens under identical conditions for any desired time duration and was used to perform comparative testing of AISI 1010 specimens treated with MIL-L-7808H/inhibitor combinations.

Another corrosion test procedure under consideration at this time involved the use of a Parr Oxygen Bomb. This procedure was evaluated under various conditions of temperature, oxygen pressure, and humidifying agents. Nominal test parameters eventually selected for oxygen-rich environmental evaluation of this procedure are shown below:

- Oxygen pressure: 0.2 MN/m^2 (2 atmospheres)
- Environmental temperature: 100°C (212°F)
- Humidity: 10 ml of 10 w/o sodium chloride in water.

The term water or deionized (DI) water refers to water with an electrical resistance of 5 to 10 megohms per centimeter.

Initially, the test duration was 3 hr, but more meaningful results were achieved when the duration was extended to 4 hr. A time/temperature equilibrium study of the Parr Bomb was run to verify the time necessary for thermal equilibrium of the test apparatus. The test chambers were modified Parr Combustion Bombs. The modification consisted of removing the straight and the looped terminals used in the firing circuitry and mounting a 1.6 mm (0.06 in.) diameter stainless steel wire across the terminal mounts in a horizontal plane. This wire has four spaced hooks to accommodate 4 panels without impingement. With this design, 4 specimens could be mounted in each test cylinder for simultaneous corrosion evaluations.

The chamber was placed in an Isotemp oven set and maintained at 100°C. Temperature readings were observed with a Fluke Digital Thermometer. Sixty-five minutes were required to attain 77°C (170°F) and 180 min to reach thermal equilibrium at 100°C, as shown in Table 1. During the 4-hr test cycle, the test specimens were maintained at 100°C during the final 60 min of the 4-hr heating cycle.

Eight tests using water adjusted to a pH of 8.0 with KOH as a humidifying agent were run early in the program (Table 2). The humidifying agent was changed from a weak potassium hydroxide to a sodium chloride solution, which appears to be a more realistic corrosive agent. The laboratory-prepared salt water used for testing was made by dissolving 10.0 g of sodium chloride (reagent grade) in 100 ml of DI water. The pH of this mixture was not controlled, but remained constant at 5.0 $\pm$ 0.2 pH.

Corrosion panels for the Parr Bomb tests were fabricated from AISI 1010 low-carbon steel in the following configuration: 2.5 x 2.5 x 0.16 cm (1.0 x 1.0 x 0.06 in.). Each panel contained a single sus-

pension hole 0.24 cm (0.09 in.) in diameter, centered 0.32 cm (0.12 in.) from the edge, midway in the panel. The panels were suspended by 1.9 cm (0.75 in.) long hooks made from 0.16 cm (0.06 in.) diameter stainless steel wire. Each test panel was identified by 0.32 cm (0.12 in.) stamped numbers located approximately 0.32 cm (0.12 in.) from the edge on the upper left side.

TABLE 1

TIME/TEMPERATURE EQUILIBRIUM STUDY OF PARR BOMB USED FOR OXYGEN-RICH CORROSION TEST

Time, min	Temperature, °C
0	26
10	36
20	45
30	54
40	62
50	69
60	75
70	79
80	83
90	86
100	88
110	90
120	93
130	94
140	95
150	97
160	98
170	99
180	100

The panels were prepared by polishing with 240 grit paper applied in the same direction across the panel. The direction of polishing was always in a horizontal plane parallel to the top edge of the panel. The edges were polished lengthwise from one corner to the next, not across the edge. The panels were held with gloves to avoid body acid/salt contamination during the polishing and cleaning procedure. When the polishing of the panel was completed, the panel was cleaned with toluene wetted cotton swabs and then immersed in boiling toluene. After all loosely adhering matter was removed from the panels, the panels were removed from the toluene, immersed in boiling acetone, removed, and allowed to flash dry before placing them in a desiccator for 30 min prior to testing.

TABLE 2

OXYGEN-RICH PRESSURIZED CORROSION TESTING

	Sample Description	Oxygen Pressure, atm	Temperature/ Humidity*	Time, hr	Panel Drain,** min/°C	Weight Change, mg	Observations
1.	Base MIL-L-7808H Oil	2	77/KOH	3	30/26	-	Moderate to severe corrosive pitting
2.	Base MIL-L-7808H Oil	2	77/KOH	3	30/26	-	Moderate to severe corrosive pitting
3.	Base MIL-L-7808H Oil	2	77/KOH	2	30/26	-	No evidence of corrosive pitting
4.	Base MIL-L-7808H Oil	1	77/KOH	2	30/26	-	Slight pitting noted
5.	Base MIL-L-7808H Oil	2	77/KOH	3	30/26	-	Slight to moderate corrosive pitting
6.	MIL-L-7808H/15 ppm PWL 80-23	2	77/KOH	3	30/26	-	Slight to moderate corrosive pitting
7.	Base MIL-L-7808H Oil	2	77/KOH	5	30/26	-	Slight to moderate corrosive pitting
8.	MIL-L-7808H/15 ppm PWL 80-23	2	77/KOH	5	30/26	-	Slight to moderate corrosive pitting
9.	Base MIL-L-7808H Oil	1	100/NaCl	3	30/26	0.0	Severe corrosive pitting
10.	Base MIL-C-8188C Oil	1	100/NaCl	3	30/26	0.0	Slight pitting noted
11.	Base MIL-L-7808H Oil	1	100/NaCl	3	15/26	-1.3	Severe corrosive pitting
12.	MIL-C-8188C Oil	1	100/NaCl	3	15/26	-0.2	No evidence of corrosive pitting
13.	Base MIL-L-7808H Oil	2	100/NaCl	6	15/26	-0.7	No evidence of corrosive pitting
14.	MIL-C-8188C Oil	2	100/NaCl	6	15/26	-0.3	No evidence of corrosive pitting
15.	Base MIL-L-7808H Oil	2	100/NaCl	3	15/26	-0.2	Medium to severe corrosive pitting
16.	MIL-C-8188C Oil	2	100/NaCl	3	15/26	-7.9	Test is void, panel fell from hook into salt water
17.	MIL-L-7808H/1.0 w/o PWL 80-63	2	100/NaCl	3	15/26	-0.1	Medium to severe corrosive pitting
18.	MIL-L-7808H/1.0 w/o PWL 80-59	2	100/NaCl	4	15/26	-0.4	Severe corrosive pitting
19.	MIL-L-7808H/1.0 w/o PWL 80-63	2	100/NaCl	4	15/26	-1.1	Moderate pitting over complete surface area of panel
20.	MIL-L-7808H/1.0 w/o PWL 80-64	2	100/NaCl	4	15/26	-1.3	Light to moderate corrosive pitting
21.	MIL-L-7808H/1.0 w/o PWL 80-65	2	100/NaCl	4	15/26	-0.9	Numerous water spots, no evidence of corrosive pitting
22.	MIL-L-7808H/1.0 w/o PWL 80-59	2	100/NaCl	4	60/100	0.0	Severe corrosive pitting
23.	MIL-L-7808H/1.0 w/o PWL 80-63	2	100/NaCl	4	60/100	-0.1	Severe corrosive pitting
24.	MIL-L-7808H/1.0 w/o PWL 80-64	2	100/NaCl	4	60/100	-0.2	Light to moderate corrosive pitting
25.	MIL-L-7808H/1.0 w/o PWL 80-65	2	100/NaCl	4	60/100	-0.2	Slight corrosive pitting, some H_2O spotting
26.	MIL-L-7808H/1.0 w/o PWL 80-60	2	100/NaCl	4	60/100	-0.4	Very severe corrosion
27.	MIL-L-7808H/1.0 w/o PWL 80-36/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	60/100	-0.1	Slight to medium corrosion
28.	MIL-L-7808H/1.0 w/o PWL 80-21	2	100/NaCl	4	Centri.	-0.1	General distribution of fine corrosive pits
29.	MIL-L-7808H/1.0 w/o PWL 80-33	2	100/NaCl	4	Centri.	0.0	General distribution of fine corrosive pits
30.	MIL-L-7808H/1.0 w/o PWL 80-34	2	100/NaCl	4	Centri.	-0.2	General distribution of fine corrosive pits
31.	MIL-L-7808H/1.0 w/o PWL 80-38	2	100/NaCl	4	Centri.	-0.1	Severe corrosive pitting
32.	MIL-L-7808H/1.0 w/o PWL 80-24	2	100/NaCl	4	Centri.	-0.1	Very slight corrosive pitting
33.	MIL-L-7808H/1.0 w/o PWL 80-26	2	100/NaCl	4	Centri.	+0.1	Light to moderate corrosive pitting
34.	MIL-L-7808H/1.0 w/o PWL 80-37	2	100/NaCl	4	Centri.	+0.3	Light to moderate corrosive pitting
35.	MIL-L-7808H/1.0 w/o PWL 80-35	2	100/NaCl	4	Centri.	+0.2	Generalized moderate corrosive pitting
36.	MIL-L-7808H/1.0 w/o PWL 80-62	2	100/NaCl	4	Centri.	0.0	Severe corrosive pitting
37.	MIL-L-7808H/1.0 w/o PWL 80-18	2	100/NaCl	4	Centri.	0.0	Medium to severe corrosive pitting
38.	MIL-L-7808H/1.0 w/o PWL 80-34	2	100/NaCl	4	Centri.	0.0	Severe corrosive pitting

TABLE 2 - Continued

OXYGEN-RICH PRESSURIZED CORROSION TESTING

	Sample Description	Oxygen Pressure, atm	Temperature/ Humidity*	Time, hr	Panel Drain,** min/°C	Weight Change, mg	Observations
39.	MIL-L-7808H/1.0 w/o PWL 80-41	2	100/NaCl	4	Centri.	0.0	Severe corrosive pitting
40.	MIL-L-7808H/1.0 w/o PWL 80-19	2	100/NaCl	4	Centri.	-0.2	Very, very fine pitting, mainly on edges
41.	MIL-L-7808H/1.0 w/o PWL 80-40	2	100/NaCl	4	Centri.	-0.1	Very slight pitting, general distribution
42.	MIL-L-7808H/1.0 w/o PWL 80-29	2	100/NaCl	4	Centri.	-0.5	Slight to moderate pitting, general distribution
43.	MIL-L-7808H/1.0 w/o PWL 80-32	2	100/NaCl	4	Centri.	0.0	Slight to moderate pitting, general distribution
44.	MIL-L-7808H/1.0 w/o PWL 80-27	2	100/NaCl	4	Centri.	-0.5	Moderate pitting, heavier around edges
45.	MIL-L-7808H/1.0 w/o PWL 80-25	2	100/NaCl	4	Centri.	-0.1	Very slight pitting, heavier around edges
46.	MIL-C-8188C Oil	2	100/NaCl	4	Centri.	-0.3	Slight pitting, heavier around edges
47.	MIL-L-7808H/1.0 w/o PWL 80-67	2	100/NaCl	4	Centri.	-0.1	Very slight pitting
48.	MIL-L-7808H/1.0 w/o PWL 80-42	2	100/NaCl	4	Centri.	+0.2	Slight to moderate corrosive pitting
49.	MIL-L-7808H/1.0 w/o PWL 80-69	2	100/NaCl	4	Centri.	0.0	Severely corroded - no good
50.	MIL-L-7808H/1.0 w/o PWL 80-28	2	100/NaCl	4	Centri.	0.0	Slight corrosive pitting, one large pit
51.	MIL-C-8188C Oil	2	100/NaCl	4	Centri.	0.0	Very slight pitting, mainly around edges
52.	MIL-L-7808H/0.7 w/o PWL 80-29/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	+0.1	Very, very fine pitting, mainly around edges
53.	MIL-L-7808H/0.7 w/o PWL 80-32/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	+0.2	Very fine pitting
54.	MIL-L-7808H/0.7 w/o PWL 80-27/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	+0.1	Fine to medium pitting, several large pits
55.	MIL-L-7808H/0.7 w/o PWL 80-42/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	+0.1	Slight pitting on one side, medium pitting on the other
56.	MIL-L-7808H/0.7 w/o PWL 80-64/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	+0.2	Very, very fine pitting, mainly around edges
57.	MIL-L-7808H/0.7 w/o PWL 80-65/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	+0.2	Slight pitting, generalized over entire panel
58.	MIL-L-7808H/0.7 w/o PWL 80-35/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	+0.1	Fine pitting
59.	MIL-L-7808H/0.7 w/o PWL 80-31/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	0.0	Medium pitting
60.	MIL-L-7808H/1.0 w/o PWL 80-20	2	100/NaCl	4	Centri.	0.0	Very, very fine pitting
61.	MIL-L-7808H/1.0 w/o PWL 80-65	2	100/NaCl	4	Centri.	-0.3	Slight to medium pitting, mainly around edges
62.	MIL-L-7808H/1.0 w/o PWL 80-39	2	100/NaCl	4	Centri.	-0.3	Slight to medium pitting
63.	MIL-L-7808H/0.7 w/o PWL 80-59/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	-0.4	Medium to severe pitting
64.	MIL-L-7808H/0.7 w/o PWL 80-63/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	-0.1	Medium pitting, mainly on edges
65.	MIL-L-7808H/0.7 w/o PWL 80-60/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	-0.2	Medium pitting, severe around edges
66.	MIL-L-7808H/0.7 w/o PWL 80-21/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	-0.5	Medium to severe pitting
67.	MIL-L-7808H/0.7 w/o PWL 80-33/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	-0.1	Medium pitting, some severity around edges

TABLE 2 - Continued

OXYGEN-RICH PRESSURIZED CORROSION TESTING

	Sample Description	Oxygen Pressure, atm	Temperature/ Humidity*	Time, hr	Panel Drain,** min/°C	Weight Change, mg	Observations
68.	MIL-L-7808H/1.0 w/o PWL 80-57	2	100/NaCl	4	Centri.	+0.7	Severely corroded - no good
69.	MIL-L-7808H/1.0 w/o PWL 80-52	2	100/NaCl	4	Centri.	+0.5	Severely corroded - no good
70.	MIL-L-7808H/1.0 w/o PWL 80-55	2	100/NaCl	4	Centri.	+0.3	Severely corroded - no good
71.	MIL-L-7808H/1.0 w/o PWL 80-53	2	100/NaCl	4	Centri.	+0.7	Severely corroded - no good
72.	MIL-L-7808H/1.0 w/o PWL 80-51	2	100/NaCl	4	Centri.	+0.5	One side very good, opposite side very slight pitting
73.	MIL-L-7808H/1.0 w/o PWL 80-56	2	100/NaCl	4	Centri.	+0.3	Slight corrosion
74.	MIL-L-7808H/0.7 w/o PWL 80-62/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	+0.3	Very, very fine pitting
75.	MIL-L-7808H/0.7 w/o PWL 80-18/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	+0.1	Very few fine spots on one edge
76.	MIL-L-7808H/0.7 w/o PWL 80-34/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	-0.7	Medium corrosive pitting
77.	MIL-L-7808H/0.7 w/o PWL 80-33/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	-0.8	Slight to medium pitting, mainly around edges
78.	MIL-L-7808H/0.7 w/o PWL 80-24/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	-0.6	Medium corrosive pitting
79.	MIL-L-7808H/0.7 w/o PWL 80-26/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	-0.6	Medium to severe corrosive pitting
80.	MIL-L-7808H/0.7 w/o PWL 80-37/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	-0.7	Severe corrosive pitting and staining
81.	MIL-L-7808H/0.7 w/o PWL 80-41/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	-0.5	Severe corrosive pitting
82.	MIL-L-7808H/0.7 w/o PWL 80-19/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	-0.8	Severe corrosive pitting
83.	MIL-C-8188C Oil	2	100/NaCl	4	Centri.	+0.7	Severe corrosive pitting plus deposits on panel
84.	MIL-L-7808H/0.7 w/o PWL 80-34/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	+0.5	Medium corrosive pitting
85.	MIL-L-7808H/0.7 w/o PWL 80-38/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	+0.6	Medium to severe corrosive pitting
86.	MIL-L-7808H/0.7 w/o PWL 80-24/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	+0.7	Medium to severe corrosive pitting
87.	MIL-L-7808H/0.7 w/o PWL 80-26/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	+1.0	Slight to medium pitting, severe around edges
88.	MIL-L-7808H/0.7 w/o PWL 80-37/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	+0.3	Slight to medium corrosive pitting
89.	MIL-L-7808H/0.7 w/o PWL 80-41/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	+0.5	Slight to medium corrosive pitting
90.	MIL-L-7808H/0.7 w/o PWL 80-19/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	+0.5	Slight corrosive pitting
91.	MIL-C-8188C Oil	2	100/NaCl	4	Centri.	+0.5	Very slight pitting one side, medium pitting on top 1/3 of reverse side

TABLE 2 - Concluded

OXYGEN-RICH PRESSURIZED CORROSION TESTING

	Sample Description	Oxygen Pressure, atm	Temperature/ Humidity*	Time, hr	Panel Drain,** min/°C	Weight Change, mg	Observations
92.	MIL-L-7808H/0.7 w/o PWL 80-23/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	+1.0	Slight to medium pitting, one side worse than the other
93.	MIL-L-7808H/0.7 w/o PWL 80-26/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	+1.1	Medium corrosive pitting
94.	MIL-L-7808H/0.7 w/o PWL 80-40/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	+1.1	Slight to medium corrosive pitting
95.	MIL-L-7808H/0.7 w/o PWL 80-25/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	+0.8	Slight corrosive pitting
96.	MIL-L-7808H/0.7 w/o PWL 80-57/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	+1.3	Medium corrosive pitting
97.	MIL-L-7808H/0.7 w/o PWL 80-52/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	+1.3	Slight to medium corrosive pitting
98.	MIL-L-7808H/0.7 w/o PWL 80-55/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	+2.4	Medium to severe corrosive pitting
99.	MIL-L-7808H/0.7 w/o PWL 80-56/ 0.3 w/o Zn Naphthenate	2	100/NaCl	4	Centri.	+1.8	Medium to severe corrosive pitting

*Temperature/Humidity Solution detail:

77/KOH 77°C (170°F), KOH in deionized H_2O, pH adjusted to 8.0

100/NaCl 100°C (212°F), 10 w/o NaCl in deionized H_2O, pH 5.0 $\pm$ 0.2

**Panel Drain detail:

30/26 30 min/26°C (79°F)

15/26 15 min/26°C (79°F)

60/100 60 min/100°C (212°F)

Centri. 30 min centrifuge at 560 rcf, ambient temperature

Three different methods of applying a test oil film on the panels were evaluated: (1) 30 sec immersion in the oil and 15 min vertical suspension at room temperature, (2) 30 min immersion in the oil and 60 min vertical suspension at 100°C, and (3) 30 sec immersion in the oil and 30 min centrifuge at room temperature at a speed to impart a relative centrifugal force (rcf) of 560 to the panels. It is believed that this last method provided the more severe test, due to the reduced oil thickness remaining on the test panels.

Ninety-nine tests utilizing the pressurized oxygen bomb procedure were run, as shown in Table 2. The same observer was used in each test. Test cycle times of 2, 3, 4, 5, and 6 hr were used to determine an optimum residence time for meaningful severity to give conclusive evidence of corrosive attack. The time/temperature study of the Parr Bomb (Table 1) indicated that a longer residence time was required to reach the desired test temperature. Therefore, the 3-hr test was replaced with a 4-hr test. A 5 or 6 hr test was deemed to be a longer test cycle than was required for meaningful data generation.

The final test procedure involved weighing the pre-polished clean specimens to $\pm$0.1 mg, oil immersion for 30 sec, centrifuging the coated specimens for 30 min, adding 10 ml salt water to the test cyclinder, and suspension of the samples in the cylinder. After sealing the cylinder, it was pressurized at 0.2 MN/m^2 oxygen and placed in an oven at 100°C. At the end of 4 hr, the cylinder was removed, vented, and the test specimens removed. The specimens were cleaned by agitation first in boiling toluene and then in boiling acetone, flash dried, and placed in a desiccator to cool for 30 min prior to weighing. After weighing, the panels were examined with a 10X magnifier. The entire area of the panel was evaluated, except for a 0.16 cm (0.06 in.) border around the four edges.

One of the two objectives of this technical effort was to establish an effective accelerated test procedure to determine the corrosion protection capability of candidate inhibitors. The oxygen-rich procedure developed for this program showed some potential for qualitative and quantitative corrosion assessment. This procedure produced meaningful data, but with considerable data scatter observed in the apparent corrosion of the same material run at different times during the course of the testing. A preferential corrosive attack on opposite sides of the same panel was found under the static test conditions of the Parr Bomb test. Further investigative work on this type of procedure was precluded because of such anomalies.

A humidity cabinet test was used to evaluate the degree of protection afforded by the oils and their protective films to metal surfaces. The length of time required to satisfactorily execute this test is prohibitive when large numbers of samples are to be studied. The method is qualitative because only visual comparisons are made between sample specimens, and allows for variations between different observers. An accelerated test procedure was also considered utiliz-

ing the ASTM B-117 Salt Spray Test in conjunction with centrifuging the sample specimens prior to testing. A series of preliminary evaluations was conducted to determine the protective capabilities of MIL-L-7808H and MIL-C-8188C with the accelerated test and to establish the test parameters necessary for comparison to humidity cabinet testing.

The humidity cabinet tests were conducted in a Precision Scientific Company Model 21174 humidity cabinet. The temperature within the test environment was maintained at 49 $\pm$ 1°C (120 $\pm$ 2°F) throughout the test. The test coupons employed in these evaluations were fabricated from AISI-1010 low carbon steel. The 3.8 X 10.2 X 0.26 cm (1.5 X 4.0 X 0.06 in.) coupons were prepared according to the procedure defined in ASTM D1748, item 7.3, using 240 grit aluminum oxide abrasive cloth. The test coupons were then cleaned by immersing in boiling toluene, followed by immersing in boiling acetone and flash drying. Test coupons that were used for more than one test were thoroughly refinished and cleaned with the same procedure prior to reuse. The test coupons were immersed in a candidate corrosion inhibiting formulation, then suspended in the humidity cabinet from suspension hooks formed from 21-gauge AMS 5837 Inconel wire. Formulations were tested with various residence times in the humidity cabinet. All post test evaluations of test coupons were conducted by the same observer. The results of these tests are shown in Table 3. The uninhibited MIL-L-7808H lubricant failed after a 24-hr exposure, while the MIL-C-8188C did not fail until after a 72-hr exposure.

Centrifuging of oil-coated specimens to reduce the thickness of the adhering oil film, thereby simulating oil film loss during long-term storage, was evaluated early in this program. Precleaned oil-coated specimens were centrifuged at 2809 rcf for time intervals of 5 min, 1 hr, and 7 hr. A significant decrease in corrosion protection was found in one hour centrifuged samples compared to 5 min samples, but the extended 7 hr centrifugation did not prove to be meaningful. Table 4 depicts MII-C-8188C protected panels without corrosion after one hour salt spray when not subjected to centrifugation, but failing in 20 min after a 5-min centrifugation and catastrophic failure after a 60 min centrifugation.

Additional test specimens were prepared and evaluated with the Salt Spray Test (ASTM B117). Preliminary tests with various salt spray exposure times showed that specimens protected by MIL-L-7808H oil failed after a 20-min exposure, and coupons protected by MIL-C-8188C failed after a 60-min exposure. It was determined in these preliminary tests that a nominal 60-min exposure to the salt spray environment produced essentially the same results as were exhibited after 72 hr in the humidity cabinet. The salt spray test, which included centrifuging the specimens for 5 min prior to the actual test, was used to evaluate approximately 35 oil/inhibitor formulations. The matrix oil was MIL-L-7808H with 1 w/o of the

TABLE 3

CORROSION PROTECTION COMPARISON OF TIME IN HUMIDITY CABINET AND CENTRIFUGE TIME

Panel Oil Film Coating Formulation	Test Cabinet Residence Time, hr	Centrifuge Time, hr	Observations
MIL-L-7808H Oil	11	None	No evidence of corrosive pitting
MIL-L-7808H Oil	11	0.250	Slight corrosive pitting
MIL-L-7808H Oil	11	1	Medium to severe corrosion
MIL-L-7808H Oil	11	7	Medium to severe corrosion
MIL-L-7808H/15 ppm PWL 80-23	11	None	No evidence of corrosive pitting
MIL-L-7808H/15 ppm PWL 80-23	11	0.250	Slight corrosive pitting
MIL-L-7808H/15 ppm PWL 80-23	11	1	Medium to severe corrosion
MIL-L-7808H/15 ppm PWL 80-23	11	7	Medium to severe corrosion
Bare unprotected panel	11	None	Severe corrosion
MIL-L-7808H Oil	24	None	Slight corrosive pitting
MIL-L-7808H/15 ppm PWL 80-23	24	None	Slight corrosive pitting
MIL-L-7808H Oil	24	0.250	Slight to medium corrosive pitting
MIL-L-7808H Oil	24	1	Medium to severe corrosion
MIL-L-7808H Oil	24	7	Medium to severe corrosion
MIL-L-7808H/15 ppm PWL 80-23	24	0.250	Slight to medium corrosive pitting
MIL-L-7808H/15 ppm PWL 80-23	24	1	Medium to severe corrosion
MIL-L-7808H/15 ppm PWL 80-23	24	7	Severe corrosion
Bare unprotected panel	24	None	Severe corrosion
MIL-L-7808H Oil	120	None	Medium to severe corrosion
MIL-L-7808H/15 ppm PWL 80-23	120	None	Medium to severe corrosion
Bare unprotected panel	120	None	Large rust spots with deep pitting

TABLE 3 - Concluded

CORROSION PROTECTION COMPARISON OF TIME IN HUMIDITY CABINET AND CENTRIFUGE TIME

Panel Oil Film Coating Formulation	Test Cabinet Residence Time, hr	Centrifuge Time, hr	Observations
MIL-L-7808H Oil	120	0.250	Severe corrosive pitting*
MIL-L-7808H/15 ppm PWL 80-23	120	0.250	Severe corrosive pitting*
MIL-L-7808H Oil	120	1	Severe corrosive pitting*
MIL-L-7808H/15 ppm PWL 80-23	120	1	Severe corrosive pitting*
MIL-L-7808H Oil	120	7	Severe corrosive pitting*
MIL-L-7808H/15 ppm PWL 80-23	120	7	Severe corrosive pitting*
MIL-C-8188C Oil	72	None	No evidence of corrosive pitting
MIL-C-8188C Oil	72	0.083	Medium to severe corrosive pitting
MIL-C-8188C Oil	72	2	Severe corrosive pitting*
MIL-L-7808H Oil	72	None	Medium corrosive pitting
MIL-L-7808H Oil	72	0.083	Medium to severe pitting*
MIL-L-7808H Oil	72	2	Severe pitting*
MIL-L-7808H/15 ppm PWL 80-23	72	None	Medium corrosive pitting
MIL-L-7808H/15 ppm PWL 80-23	72	0.083	Medium to severe pitting*
MIL-L-7808H/15 ppm PWL 80-23	72	2	Severe pitting*
MIL-L-7808H/1.0 w/o PWL 80-23	72	None	Slight corrosive pitting
MIL-L-7808H/1.0 w/o PWL 80-23	72	0.083	Slight to medium corrosive pitting
MIL-L-7808H/1.0 w/o PWL 80-23	72	1	Severe corrosive pitting*

*Entire panel area

TABLE 4

ASTM B-117 SALT SPRAY CORROSION EVALUATION - A COMPARISON OF SALT SPRAY TIME AND CENTRIFUGE TIME

Panel Oil Film Coating Formulation	Salt Spray Exposure Time, min	Centrifuge Time, min	Observations
MIL-C-8188C Oil	20	None	No evidence of corrosive pitting
MIL-C-8188C Oil	20	5	Slight to medium corrosive pitting
MIL-C-8188C Oil	20	60	Medium to severe corrosive pitting
MIL-C-8188C Oil	60	None	No evidence of corrosive pitting
MIL-C-8188C Oil	60	5	Slight to medium corrosive pitting
MIL-C-8188C Oil	60	60	Severe corrosive pitting
MIL-L-7808H Oil	20	None	Slight to medium corrosive pitting
MIL-L-7808H Oil	20	5	Severe corrosive pitting
MIL-L-7808H Oil	20	60	Severe corrosive pitting*
MIL-L-7808H Oil	60	None	Medium to severe corrosive pitting
MIL-L-7808H Oil	60	5	Severe corrosive pitting
MIL-L-7808H Oil	60	60	Severe corrosive pitting*
MIL-L-7808H/15 ppm PWL 80-23	20	None	Slight to medium corrosive pitting
MIL-L-7808H/15 ppm PWL 80-23	20	5	Severe corrosive pitting
MIL-L-7808H/15 ppm PWL 80-23	20	60	Severe corrosive pitting*

*Entire panel area

various additives. The initial screening test utilized an exposure of one hour of the oil/inhibitor-coated specimen, as shown in Table 5. Comparitive evaluations of various corrosion inhibiting oil formulations indicated that the better or more corrosion resistent formulations provided a longer life expectancy in both media.

A Potentiodyne Analyzer was evaluated as a method for rapid determination of the electrochemical measurements of corrosion rate. The sensitivity and application of the instrument have been evaluated. Initial test cell design and procedural analysis indicated that the development of this instrument for corrosion measurement was beyond the scope of this program because of the need to also develop a compatible electrolyte.

Much technical effort was spent in the final development and validation of the accelerated Corrosion Rate Evaluation Procedure (CREP) for use in screening the corrosion inhibiting characteristics of candidate corrosion inhibitors. As previously mentioned, the CREP utilizes a buffered acetate solution at 100°C with a dynamic airflow system maintained at a flowrate of 45 cc/min. This procedure has been written in standard form as given by the American Society for Testing and Materials (ASTM) and is included herein as Appendix A, along with supplementary notes concerning use with MIL-L-7808H type engine oils.

During this period of development, several problems were encountered and eliminated. The first of these involved the development of a repeatable method of surface preparation for the specimen. It was found that significant differences existed between the samples using identical reaction kettles, and even within the same kettle. This problem was reduced, but not eliminated, by the development of a new cleaning procedure. Another problem occurred because a film developed on specimens in the acetic acid buffers when the molar concentration was greater than 0.5. The test procedure was modified to allow a maximum molar concentration of 0.1 in total acetate in order to eliminate this problem. A further problem was found to occur with two of the three types of rubber stoppers used to support the specimens. The acetic buffer solution attacked the exposed surface of the stopper and resulted in deterioration of the stopper. Buna-N stoppers were found to be compatible with the acetic acid vapors under the test conditions described herein.

Contractual obligations stipulated that 10 fluid samples furnished by the AFWAL/MLBT Program Manager be evaluated for corrosion protection. These fluids were to be ranked as more effective, less effective, or equivalent to MIL-C-8188C oil in corrosion protection.

Prior to receipt of these samples, 15 CREP analyses were conducted comparing corrosion protection of MIL-C-8188C to MIL-L-7808H oil, as shown in Table 6. The data reveals that the CREP analysis is capable of repeatedly differentiating the corrosion protection furnished by the two oils.

TABLE 5

EFFECTS OF ONE HOUR SALT SPRAY CORROSION TESTING
USING 1.0% ADDITIVE/MIL-L-7808H OIL MIXTURES

	1.0 w/o Additive	Panel No.	Observations
1.	Base MIL-L-7808H Oil	77	Dark spots covering surfaces
2.	PWL 80-43	83	General distribution of spots
3.	PWL 80-66	85	Small spots
4.	PWL 80-60	86	Spots, not evenly distributed over surface
5.	PWL 80-68	87	Surface area covered with spots
6.	PWL 80-62	90	Small spots distributed over entire surface
7.	PWL 80-18	91	Very slight number of small spots appearing
8.	PWL 80-59	92	Surface covered with spots
9.	PWL 80-20	93	Very slight number of small spots appearing
10.	PWL 80-65	114	Surface covered with spots
11.	PWL 80-37	139	Small spots distributed over entire surface
12.	PWL 80-24	140	Even distribution of spots
13.	PWL 80-27	141	Spotting, not evenly distributed
14.	PWL 80-30	142	Spotting, not evenly distributed
15.	PWL 80-34	143	Areas of small spots
16.	PWL 80-23	144	Top of panel corroded
17.	PWL 80-42	145	Panel covered with spots
18.	PWL 80-41	146	Spotting, not evenly distributed
19.	PWL 80-69	147	Large streaks, remainder of panel clear
20.	PWL 80-19	148	Small spots
21.	PWL 80-31	149	Spotting, not evenly distributed
22.	PWL 80-34	150	Very small spots
23.	PWL 80-40	151	Small to medium sized spots
24.	PWL 80-26	152	Panel surface covered with spots
25.	PWL 80-28	135	Small spots covering panel
26.	PWL 80-33	136	Panel covered with rust
27.	PWL 80-21	137	Infrequent isolated spotting
28.	PWL 80-67	154	Spotted
29.	PWL 80-61	155	Spotted
30.	PWL 80-39	156	Spotted
31.	PWL 80-63	157	Covered with small spots
32.	0.7 w/o PWL 80-31/0.3 w/o Zinc Naphthenate	158	Irregular areas of rust
33.	0.7 w/o PWL 80-42/0.3 w/o Zinc Naphthenate	159	Panel surface covered by rust
34.	0.7 w/o PWL 80-35/0.3 w/o Zinc Naphthenate	162	Small spots appearing
35.	0.7 w/o PWL 80-65/0.3 w/o Zinc Naphthenate	163	Panel surface covered by spots

TABLE 6

CORROSION RATE TESTS CONDUCTED TO DETERMINE THE REPEATABILITY OF THE CORROSION RATE EVALUATION PROCEDURE

	Corrosion Rates,* mg/hr		Difference in Corrosion Rates,* mg/hr
	MIL-C-8188C	MIL-L-7808H, QRN 15F-1	
1.	10.7	16.3	5.6
2.	14.7	20.8	6.1
3.	8.8	15.3	6.5
4.	11.3	17.8	6.5
5.	8.5	14.0	5.5
6.	10.2	16.6	6.4
7.	8.4	14.7	6.3
8.	8.0	15.3	7.3
9.	7.2	14.4	7.2
10.	7.3	14.6	7.3
11.	8.6	14.2	5.6
12.	9.1	15.6	6.5
13.	8.9	15.6	6.7
14.	9.3	16.1	6.8
15.	8.2	14.5	6.3

*All corrosion rate (CR) data represent the weight loss of 5.08 x 1.27 x 0.159 cm (2.0 x 0.5 x 0.0625 in.) AISI-1010 steel coupons, in mg/hr. The difference in corrosion rates is equal to the CR with MIL-L-7808H protection, minus the CR with MIL-C-8188C protection.

Ten fluid samples were furnished by the AFWAL/MLBT Program Manager for validation of the P&WA/GPD-developed CREP and were evaluated for their corrosion protection capabilities relative to MIL-C-8188C. The results of these analyses were forwarded to the AFWAL/MLBT Program Manager, who has reviewed the data concerning these validation samples and responded with a letter discussing the results of the tests. The Air Force has accepted the CREP as an adequate preliminary screening test. P&WA/GPD is currently initiating effort in an endeavor to increase the precision and repeatability of the CREP at the request of the AFWAL/MLBT Program Manager.

SECTION IV

TASK 4.3 - FORMULATION AND EVALUATION OF CORROSION INHIBITING ENGINE OILS

Three different samples of MIL-L-7808H aircraft turbine engine lubricants were acquired to formulate the candidate corrosion inhibiting engine oils to be evaluated during this program. These lubricant formulation stocks were selected from the MIL-L-7808H Qualified Products List (QPL). The acquisition of three oil samples was based on the fact that differences in composition will exist in the qualified formulations from different manufacturers. These compositional variations in the qualified lubricants may contribute to misleading conclusions due to the possible interaction with the various organic corrosion inhibiting compounds. For example, a given inhibitor may result in excellent corrosion protection in one oil, but may be much less effective in another oil conforming to the same specification requirements. Therefore, three MIL-L-7808H oil formulations were obtained in order to evaluate the relative increase in corrosion protection afforded through the addition of the most promising candidate corrosion inhibitors (CCI).

All of the formulations evaluated thus far in this investigation were blended with a MIL-L-7808H formulation having a QPL designation 15F-1, manufactured under the Mobil Chemical Company Quality Reference Number, QRN 15F-1.[1] The selection of the QRN 15F-1 formulation was predicated on its relatively low volatility and evaporation rate. This is important due to the necessity to minimize the evaporation rate of the Air-Launched Cruise Missile (ALCM) corrosion inhibiting engine oil (Reference 3). Future evaluations to verify the effectiveness of the most promising CCI will include analyses performed on formulations blended with MIL-L-7808H having QPL designations 11E-1 and 15E-1.

During the first phase of this program, a major effort was directed toward the acquisition of samples of commercially available corrosion inhibitors. In addition, 16 CCI resulted from the subcontracted custom synthesis by Bray Oil Company. Twenty manufacturers and vendors were contacted during the industry search, resulting in the acquisition of 51 CCI samples at no charge to the program. This brought the total number of CCI to 67, and included representatives of several classes of corrosion inhibiting compounds. The inhibitor manufacturers that supplied samples of CCI are listed in the acknowledgements.

A general description of the active ingredients (AI) of each CCI evaluated in this program is provided in Table 7. Many inhibitors contained metal sulfonates as their AI. These included monovalent and divalent salts of various alkylated benzene sulfonic acids, in addition to amine salts of sulfonic acids. Other polar inhibitors evaluated included primary and secondary amines, organic acids, esters,

1. Unless otherwise stated, all references to MIL-L-7808H oil or oil/inhibitor formulations will indicate Mobil Chemical Company MIL-L-7808H oil with a QRN of 15F-1.

TABLE 7

A GENERAL DESCRIPTION OF THE ACTIVE INGREDIENT
OF EACH CANDIDATE CORROSION INHIBITOR

Inhibitor Code Number	Active Ingredient Description	Active Ingredient w/o
PWL 80-1	Mixture of calcium sulfonate and polyglycol	31
PWL 80-2	Calcium sulfonate from mineral oil	40
PWL 80-3	Mixture of calcium sulfonate and calcium carbonate	30
PWL 80-4	Slightly basic calcium sulfonate from mineral oil	40
PWL 80-5	Morpholine sulfonate plus dialkyl benzene sulfonate	100
PWL 80-6*	Zinc sulfonate from alkylated (C_{30}) benzene	45
PWL 80-7*	Calcium sulfonate from alkylated (C_{30}) benzene	44
PWL 80-8*	Barium sulfonate from alkylated (C_{30}) benzene	46
PWL 80-9*	Magnesium sulfonate from alkylated (C_{30}) benzene	43
PWL 80-10*	Potassium sulfonate from alkylated (C_{45}) benzene	29
PWL 80-11*	Lithium sulfonate from alkylated (C_{45}) benzene	28
PWL 80-12*	Calcium sulfonate from alkylated (C_{45}) benzene	31
PWL 80-13*	Barium sulfonate from alkylated (C_{45}) benzene	31
PWL 80-14*	Calcium sulfonate from alkylated (C_{30}) benzene	44
PWL 80-15*	Barium sulfonate from alkylated (C_{30}) benzene	47
PWL 80-16*	Barium sulfonate from alkylated (C_{20}) benzene	30
PWL 80-17	Mixture of organic acids	75
PWL 80-18	Alkyl ammonium - alkyl acid phosphate	100
PWL 80-19	Amine neutralized organic acid	78
PWL 80-20	Organic acid	75
PWL 80-21	Alkyl ammonium - alkyl phosphate	80
PWL 80-22	Mixture of organic acid and organic acid phosphate	50
PWL 80-23	Mixture of alkyl succinic acids and esters	63
PWL 80-24	Amine, amid imidazoline product from fatty acids	75
PWL 80-25	Alkyl succinic acid	63
PWL 80-26	Alkyl succinic acid plus hydroxylated alkyl phenol	89
PWL 80-27	Calcium sulfonate	40
PWL 80-28	Amine salt of an alkyl succinic acid	61
PWL 80-29	Alkyl acid ester - alkyl succinic anhydride	63
PWL 80-30	Slightly basic calcium sulfonate	43
PWL 80-31	Sodium sulfonate	60
PWL 80-32	Alkyl succinic acid	61
PWL 80-33	Phosphoric acid	100
PWL 80-34	Zinc salt of a carboxylated alkyl phenol	54
PWL 80-35	High molecular weight alkyl succinic acid	100
PWL 80-36	Hydroxylated alkyl phenol plus a zinc salt of a carboxylated alkyl phenol	60

TABLE 7 - Concluded

A GENERAL DESCRIPTION OF THE ACTIVE INGREDIENT
OF EACH CANDIDATE CORROSION INHIBITOR

Inhibitor Code Number	Active Ingredient Description	Active Ingredient w/o
PWL 80-37	Barium sulfonate	56
PWL 80-38	Barium sulfonate	51
PWL 80-39	Mixture of sulfurized alkyl phenol and alkyl succinic acid	82
PWL 80-40	Mixture of hydroxy ethyl alkyl phenol, sulfurized alkyl phenol, and alkyl succinic anhydride	82
PWL 80-41	Polycarboxylic acid salt of a fatty acid/polyamine reaction product	50
PWL 80-42	Mixture of polycarboxylic acids	50
PWL 80-43	Amine neutralized phosphoramidate/alkyl phosphate	80
PWL 80-44	Imidazoline	100
PWL 80-45	Cyclic amine salt of a polycarboxylic acid	63
PWL 80-46**	Secondary amine	100
PWL 80-47**	Secondary amine	100
PWL 80-48**	Primary amine	100
PWL 80-49**	Fluorinated carboxylic acid	100
PWL 80-50**	Fluorinated quaternary ammonium salt	100
PWL 80-51**	Fluorinated sulfonamide	100
PWL 80-52**	Fluorinated alcohol	100
PWL 80-53	Polyalkoxylated fluorinated alcohol	100
PWL 80-54**	Fluorinated carboxylic acid	100
PWL 80-55**	Fluorinated alcohol	100
PWL 80-56**	Fluorinated alcohol	100
PWL 80-57**	Fluorinated sulfonamide	100
PWL 80-58**	Fluorinated carboxylic acid	100
PWL 80-59	Polyamino-alcohol	20
PWL 80-60	Mixture of amine neutralized dimer acids and amine phosphate esters	50
PWL 80-61	Polyamino-alcohol	50
PWL 80-62	Polyamino-alcohol	50
PWL 80-63	Amine dimer	20
PWL 80-64	Amine phosphate ester	50
PWL 80-65	Amine neutralized dimer acid	60
PWL 80-66	Dimer acid	50
PWL 80-67	Mixture of amine neutralized dimer acid and phosphate esters	40

*The average length of the alkyl chains is indicated parenthetically.
**These candidate corrosion inhibitors (CCI) were received as solids.

alkyl phosphates, and various fluorinated alcohols and acids. Several of these organic inhibitors were received as solids. Table 7 also provides the concentration of AI in each of the CCI. Many of the inhibitors were received in kerosene, mineral oil, or aromatic solvents. The percentage of AI in the CCI varied from 20 to 100 weight percent (w/o).

Concurrent with communications to obtain the information presented in Table 7, preliminary evaluations were conducted using formulations containing 1.0 w/o of the CCI "as received" from the manufacturer.[2] For example, a 5g aliquot of the inhibitor "as received" was diluted with 495g of MIL-L-7808H. The corrosion rate data generated in these tests are presented in Table 8. The w/o of the AI in each formulation is listed for comparison. These corrosion rates were determined with the P&WA/GPD-developed CREP defined in Appendix A. All corrosion rates in this table represent the weight loss in mg/hr of 5.08 x 1.27 x 0.16 cm (2.0 x 0.5 x 0.06 in.) AISI 1010 sample coupons. The difference in corrosion rates equals the corrosion rate of the coupon coated with MIL-L-7808H/inhibitor minus the corrosion rate of the coupon coated with MIL-C-8188C in the same test. Therefore, the smaller the differential corrosion rate, the more effective the CCI.

As shown by the data in Table 8, none of the inhibitors was found to be more effective than MIL-C-8188C at 1.0 w/o "as received." Only two of the CCI evaluated at this concentration provided corrosion protection which was nearly equivalent to the protection afforded by MIL-C-8188C. Furthermore, it was observed that some of the CCI appeared to impair the inherent corrosion protection of MIL-L-7808H, resulting in corrosion rates in excess of those observed for uninhibited MIL-L-7808H. For example, tests conducted with coupons protected by PWL 80-60 resulted in a differential corrosion rate of 9.6 mg/hr. Similar results were observed in tests conducted with PWL 80-51 and 80-55. As shown in Table 6, the average difference in corrosion rates between coupons protected by uninhibited MIL-L-7808H and MIL-C-8188C was approximately 6.5 mg/hr.

In order to provide a more valid comparison of the CCI, all evaluations subsequent to the acquisition of the information in Table 7 involved formulations blended on the basis of the AI concentration of CCI in MIL-L-7808H. Technical discussions were held with lubricating oil and CCI manufacturers in order to establish a realistic range of AI concentrations to be used in evaluating the CCI. A wide range of corrosion inhibitor concentrations was found to exist in current applications. These varied from 0.1 to 10.0 w/o AI and were found to be dependent upon the intended application of the formulation. Based on the preliminary tests performed at 1.0 w/o "as received" in which none of the inhibitors proved more effective than MIL-C-8188C, a minimum concentration of 0.5 w/o AI was established for this investigation. A maximum concentration of 2.0 w/o AI was selected, since this would involve the addition of up to 10 w/o of some CCI "as received." Greater concentrations were considered prohibitive based on the need

2. Percentage by weight "as received" denotes the percent of the total product, which includes the AI and the carrier.

TABLE 8

DATA FROM THE CORROSION RATE EVALUATION PROCEDURE
FOR CANDIDATE CORROSION INHIBITORS (CCI) AT 1.0 w/o* IN MOBIL MIL-L-7808H, QRN 15F-1

Inhibitor Code Number	Concentration of AI in MIL-L-7808H, w/o**	Corrosion Rates,*** mg/hr		Difference in Corrosion Rates,*** mg/hr
		MIL-C-8188C	MIL-L-7808H Plus 1.0 w/o CCI*	
1. PWL 80-32	0.61	8.0	8.7	0.7
		9.8	9.5	-0.3
		8.9	10.8	1.9
2. PWL 80-29	0.63	9.8	10.3	0.5
		10.6	11.4	0.8
		10.0	10.5	0.5
		9.6	11.3	1.7
3. PWL 80-20	0.75	8.8	10.2	1.4
		9.4	11.6	2.2
4. PWL 80-33	1.00	9.0	11.1	2.1
5. PWL 80-21	0.80	9.8	12.1	2.3
6. PWL 80-18	1.00	7.2	8.5	1.3
		8.1	10.6	2.5
7. PWL 80-35	1.00	8.1	10.9	2.8
		7.6	10.6	3.0
		7.1	10.6	3.5
8. PWL 80-2	0.40	8.7	11.9	3.2
9. PWL 80-31	0.60	8.6	11.9	3.3
10. PWL 80-25	0.63	9.5	13.3	3.8
11. PWL 80-23	0.63	7.5	11.3	3.8
12. PWL 80-64	0.50	7.5	11.3	3.8
		8.0	13.1	5.1
		8.4	12.3	3.9
13. PWL 80-4	0.40	7.7	12.4	4.7
14. PWL 80-17	0.75	6.9	12.5	5.6
		8.0	12.6	4.6
15. PWL 80-43	0.50	9.8	14.2	4.4
16. PWL 80-38	0.51	8.0	13.3	5.3
		9.7	15.8	6.1
17. PWL 80-65	0.60	9.5	15.3	5.8
18. PWL 80-40	0.82	7.9	13.3	5.4
19. PWL 80-34	0.54	9.7	15.5	5.8
20. PWL 80-39	0.82	8.3	14.3	6.0

TABLE 8 - Concluded

DATA FROM THE CORROSION RATE EVALUATION PROCEDURE
FOR CANDIDATE CORROSION INHIBITORS (CCI) AT 1.0 w/o* IN MOBIL MIL-L-7808H, QRN 15F-1

Inhibitor Code Number	Concentration of AI in MIL-L-7808H, w/o**	Corrosion Rates,*** mg/hr		Difference in Corrosion Rates,*** mg/hr
		MIL-C-8188C	MIL-L-7808H Plus 1.0 w/o CCI*	
21. PWL 80-37	0.56	9.0	15.3	6.3
22. PWL 80-30	0.43	7.7	13.8	6.1
23. PWL 80-19	0.78	10.2	16.4	6.2
24. PWL 80-28	0.61	8.2	15.1	6.9
25. PWL 80-48	1.00	9.0	15.7	6.7
26. PWL 80-67	0.40	9.5	15.9	6.4
		9.1	17.2	8.1
		8.2	14.7	6.5
27. PWL 80-27	0.40	9.2	14.1	4.9
		9.3	16.3	7.0
		9.1	16.3	7.2
28. PWL 80-26	0.89	10.2	17.1	6.9
29. PWL 80-1	0.31	8.2	15.2	7.0
30. PWL 80-47	1.00	9.4	16.8	7.4
		10.0	17.2	7.2
31. PWL 80-41	0.50	7.0	14.1	7.1
32. PWL 80-56	1.00	10.6	18.3	7.7
		9.8	19.3	9.5
33. PWL 80-52	1.00	9.7	16.7	7.0
34. PWL 80-36	0.60	7.3	15.7	8.4
		7.9	16.2	8.3
35. PWL 80-46	1.00	7.4	15.5	8.1
		9.0	17.0	8.0
36. PWL 80-55	1.00	9.3	18.2	8.9
37. PWL 80-60	1.00	9.6	19.2	9.6
38. PWL 80-51	1.00	9.1	17.8	8.7

*w/o of CCI as received from manufacturer

**The active ingredient (AI) of CCI in MIL-L-7808H is calculated on the basis of the w/o of AI in the CCI "as received."

***All corrosion rate (CR) data represent the weight loss of 5.08 x 1.27 x 0.159 cm (2.0 x 0.5 x 0.0625 in.) AISI-1010 steel coupons, in mg/hr. The difference in CR is equal to the CR with MIL-L-7808H plus CCI, minus the CR with MIL-C-8188C.

to preserve the physical and chemical properties of the MIL-L-7808H oil.

In order to accomplish the evaluation of 67 CCI, a screening test was developed that involved the sequential elimination of the less desirable CCI on the basis of their solubility and corrosion inhibiting characteristics. This test matrix provided a means of selecting the most promising CCI for a more in-depth analysis of their corrosion protection capabilities at various concentrations, in addition to their effect on the physical and chemical properties of the base MIL-L-7808H lubricating oil. After establishing the maximum and minimum AI concentrations for this investigation, the initial screening of the 67 CCI was performed on the basis of their solubility characteristics in MIL-L-7808H oil. Predicated on the importance of eliminating the potential for sludge formation during long-term storage of the corrosion inhibiting engine oil, any candidate formulations which exhibited measurable sedimentation in the solubility tests were rejected. The oil/inhibitor formulations were blended using a magnetic stirring hot plate. Many of the CCI blended easily at room temperature, 25°C (77°F). Those CCI that were difficult to formulate in the MIL-L-7808H at room temperature were heated to a maximum temperature of 68°C (155°F) in conjunction with magnetic stirring. This temperature was selected to preclude any thermal degradation of the oil/inhibitor formulation during the blending procedure.

These formulations were subsequently subjected to a 30 min centrifugation at 560 rcf. This provided an accelerated method of determining the extent of dissolution of the CCI by concentrating sludges, precipitates, or colloidal suspensions. After recording the appearance of each formulation, a 100 ml aliquot was centrifuged for 30 min. The results of these tests are presented in Table 9, which gives the formulation appearance and the extent of sedimentation after the centrifugation. This table also includes the method employed in blending each CCI with the MIL-L-7808H oil.

Of the 67 CCI, 21 were found to be immiscible or insoluble at 2.0 w/o AI. The CCI that did not result in homogeneous solutions after 30 min of magnetic stirring at 68°C were rejected without centrifugation. Three CCI exhibited no visible stratification after centrifugation, but showed significant sedimentation during room temperature storage for 1 to 10 days. Therefore, predicated on the importance of eliminating the potential for sludge formation in the corrosion inhibiting oil, these CCI were rejected at 2.0 w/o AI. The superscripts in Table 9 indicate which CCI were rejected in this phase of the screening test series.

The 21 CCI that were immiscible at the maximum concentration of 2.0 w/o AI were subsequently evaluated at the minimum concentration of 0.5 w/o AI. These CCI were formulated with MIL-L-7808H using the procedure previously described for the miscibility tests. The formulations were then centrifuged and evaluated on the basis of sedimentation during centrifugation. The data generated in these tests is presented in Table 10. Of the 21 CCI assessed at 0.5 w/o AI, 16 were found to be immiscible. Consequently, these 16 CCI were rejected from

TABLE 9

SOLUBILITY CHARACTERISTICS OF THE CANDIDATE CORROSION INHIBITORS
AT 2.0 w/o ACTIVE INGREDIENT IN MOBIL MIL-L-7808H, QRN 15F-1

	Inhibitor Code Number	Formulation Appearance	Formulating Method*	Formulation Appearance After 30 min Centrifugation at 560 rcf
1.	PWL 80-1	Clear, miscible	1	No visible stratification
2.	PWL 80-2	Slightly darkened, miscible	1	No visible stratification
3.	PWL 80-4	Slightly darkened, miscible	1	No visible stratification
4.	PWL 80-6	Dark, miscible	2	No visible stratification
5.	PWL 80-7	Slightly darkened, miscible	1	No visible stratification
6.	PWL 80-8	Dark, miscible	2	No visible stratification
7.	PWL 80-10	Slightly darkened, miscible	1	No visible stratification
8.	PWL 80-11	Dark, miscible	1	No visible stratification
9.	PWL 80-12	Slightly darkened, miscible	1	No visible stratification
10.	PWL 80-14	Dark, miscible	1	No visible stratification
11.	PWL 80-17	Clear, miscible	1	No visible stratification
12.	PWL 80-18	Clear, miscible	1	No visible stratification
13.	PWL 80-20	Clear, miscible	1	No visible stratification
14.	PWL 80-21	Clear, miscible	1	No visible stratification
15.	PWL 80-22	Dark, miscible	1	No visible stratification
16.	PWL 80-23	Clear, miscible	1	No visible stratification
17.	PWL 80-25	Clear, miscible	1	No visible stratification
18.	PWL 80-26	Clear, miscible	1	No visible stratification
19.	PWL 80-28	Turbid	2	No visible stratification
20.	PWL 80-29	Clear, miscible	1	No visible stratification
21.	PWL 80-30	Clear, miscible	1	No visible stratification
22.	PWL 80-32	Clear, miscible	1	No visible stratification
23.	PWL 80-33	Clear, miscible	1	No visible stratification
24.	PWL 80-34	Dark, miscible	1	No visible stratification
25.	PWL 80-35	Clear, miscible	1	No visible stratification
26.	PWL 80-36	Dark, miscible	1	No visible stratification
27.	PWL 80-37	Dark, miscible	1	No visible stratification
28.	PWL 80-38	Dark, miscible	1	No visible stratification
29.	PWL 80-39	Dark, miscible	1	No visible stratification
30.	PWL 80-40	Dark, miscible	1	No visible stratification
31.	PWL 80-42	Clear, miscible	1	No visible stratification
32.	PWL 80-43	Clear, miscible	1	No visible stratification
33.	PWL 80-44	Clear, miscible	1	No visible stratification
34.	PWL 80-46[b]	Clear, soluble	2	No visible stratification
35.	PWL 80-47[b]	Clear, soluble	3	No visible stratification
36.	PWL 80-48[b]	Clear, soluble	2	No visible stratification
37.	PWL 80-51[b]	Clear, soluble	2	No visible stratification
38.	PWL 80-52[b]	Clear, soluble	2	No visible stratification
39.	PWL 80-53	Clear, miscible	2	No visible stratification
40.	PWL 80-55[b]	Clear, soluble	2	No visible stratification
41.	PWL 80-56[b]	Clear, soluble	2	No visible stratification
42.	PWL 80-59	Clear, miscible	1	No visible stratification
43.	PWL 80-62	Clear, miscible	1	No visible stratification
44.	PWL 80-64	Clear, miscible	1	No visible stratification

TABLE 9 - Concluded

SOLUBILITY CHARACTERISTICS OF THE CANDIDATE CORROSION INHIBITORS
AT 2.0 w/o ACTIVE INGREDIENT IN MOBIL MIL-L-7808H, QRN 15F-1

	Inhibitor Code Number	Formulation Appearance	Formulating Method*	Formulation Appearance After 30 min Centrifugation at 560 rcf
45.	PWL 80-24	Turbid	2	Minute amount of brown sediment
46.	PWL 80-31	Turbid	2	Minute amount of brown sediment
47.	PWL 80-9[c]	Dark, creamy deposit formed after 9 days	2	No visible stratification
48.	PWL 80-13[c]	Turbid, creamy deposit formed after 24 hr	2	No visible stratification
49.	PWL 80-15[c]	Turbid, creamy deposit formed after 24 hr	2	No visible stratification
50.	PWL 80-3[a]	Dark, very turbid, not homogeneous	2	No centrifugation
51.	PWL 80-5[a]	Dark, not homogeneous	2	No centrifugation
52.	PWL 80-16[a]	Very turbid	2	4.6 ml creamy sediment
53.	PWL 80-19[a]	Slightly turbid, not homogeneous	2	No centrifugation
54.	PWL 80-27[a]	Slightly turbid, not homogeneous	1	0.1 ml white sediment
55.	PWL 80-41[a]	Dark, turbid, not homogeneous	2	0.1 ml dark brown deposit, streaks of brown deposited on tube walls
56.	PWL 80-45[a]	Dark, turbid	2	1.6 ml dark brown deposit
57.	PWL 80-49[a,b]	Very turbid, not homogeneous	2	No centrifugation
58.	PWL 80-50[a,b]	Turbid, not homogeneous	2	No centrifugation
59.	PWL 80-54[a,b]	Very turbid, not homogeneous	2	No centrifugation
60.	PWL 80-57[a,b]	Turbid, not homogeneous	2	No centrifugation
61.	PWL 80-58[a,b]	Clear, not homogeneous	2	No centrifugation
62.	PWL 80-60[a]	Dark, turbid, not homogeneous	2	No centrifugation
63.	PWL 80-61[a]	Very turbid, not homogeneous	2	No centrifugation
64.	PWL 80-63[a]	Turbid, not homogeneous	2	No centrifugation
65.	PWL 80-65[a]	Very dark, not homogeneous	2	No centrifugation
66.	PWL 80-66[a]	Dark, turbid, not homogeneous	2	No centrifugation
67.	PWL 80-67[a]	Turbid, not homogeneous	1	No centrifugation

[a]These candidate corrosion inhibitors (CCI) were rejected at 2.0 w/o active ingredient (AI) and blended at 0.5 w/o AI in Mobil MIL-L-7808H, QRN 15F-1.

[b]These CCI were received as solids.

[c]These CCI appeared to be miscible, and exhibited no visible stratification after centrifugation; however, the formation of precipitate was evident after 1 to 10 days storage. These CCI were rejected at 2.0 w/o AI and blended at 0.5 w/o AI.

*Key to Formulating Method:

1. Magnetic Stirring, 25°C (77°F)
2. Magnetic Stirring, 68°C (155°F) maximum temperature
3. Solid sample of CCI was melted at 63°C (145°F) and mixed to ensure homogeneity prior to taking aliquot for formulating with MIL-L-7808H, QRN 15F-1.

TABLE 10

SOLUBILITY CHARACTERISTICS OF THE CANDIDATE CORROSION INHIBITORS
(PREVIOUSLY REJECTED AT MAXIMUM CONCENTRATION)
AT 0.5 w/o ACTIVE INGREDIENT IN MOBIL MIL-L-7808H, QRN 15F-1

Inhibitor Code Number	Formulation Appearance	Formulating Method*	Formulation Appearance After 30 min Centrifugation at 560 rcf
1. PWL 80-15	Clear, miscible	2	No visible stratification
2. PWL 80-16	Slightly turbid, miscible	1	No visible stratification
3. PWL 80-27	Clear, miscible	1	No visible stratification
4. PWL 80-57[b]	Clear, soluble	2	Minute amount of brown deposit
5. PWL 80-19	Slightly turbid, miscible	2	Minute light brown deposits on the walls
6. PWL 80-3[a]	Dark, turbid, not homogeneous	2	0.7 ml light brown deposit
7. PWL 80-5[a]	Clear, not homogeneous	2	No centrifugation
8. PWL 80-9[c]	Slightly turbid, miscible	2	No visible stratification
9. PWL 80-13[c]	Slightly turbid	1	No visible stratification
10. PWL 80-41[a]	Turbid, not homogeneous	2	No centrifugation
11. PWL 80-45[a]	Turbid, not homogeneous	2	No centrifugation
12. PWL 80-58[a,b]	Slightly turbid, not homogeneous	2	No centrifugation
13. PWL 80-54[a,b]	Slightly turbid, not homogeneous	2	No centrifugation
14. PWL 80-49[a,b]	Turbid, not homogeneous	2	No centrifugation
15. PWL 80-50[a,b]	Turbid, not homogeneous	2	No centrifugation
16. PWL 80-63[a]	Turbid, not homogeneous	2	No centrifugation
17. PWL 80-65[a]	Dark, not homogeneous	2	No centrifugation
18. PWL 80-66[a]	Dark, not homogeneous	2	No centrifugation
19. PWL 80-67[a]	Turbid	2	Dark deposits on centrifuge tube walls
20. PWL 80-61[a]	Turbid	2	0.4 ml cloudy white deposit
21. PWL 80-60[a]	Slightly turbid, not homogeneous	2	No centrifugation

[a]These candidate corrosion inhibitors (CCI) were rejected from this investigation because of their tendency to form sludges or precipitates at the maximum and minimum concentrations. Those formulations that were not homogeneous after 30 minutes of magnetic stirring at 68°C (155°F) were rejected without centrifugation.

[b]These CCI were received as solids.

[c]These CCI appeared miscible upon formulation and exhibited no visible stratification after centrifugation; however, they formed precipitates after storage for 1-10 days. Therefore, these CCI were rejected from the investigation of single additive formulations.

*Key to Formulating Method:

1. Magnetic Stirring, 25°C (77°F)

2. Magnetic Stirring, 68°C (155°F) maximum temperature

the investigation of single additive formulations based on their immiscibility at the minimum AI concentration. The superscripts in Table 10 indicate which CCI were rejected.

The next phase of the screening test series involved evaluating the relative corrosion inhibiting capabilities of the 46 CCI that were miscible at 2.0 w/o AI. The corrosion tests used to assess these oil/inhibitor formulations were performed according to the CREP defined in Appendix A and the Sequential Sampling Plan (SSP) defined in Appendix B. These tests defined the corrosion protection provided by a thin film of each formulation, relative to the protection provided by MIL-C-8188C. The SSP was developed to expedite the elimination of the less effective CCI with 95% confidence.

The SSP was predicated on the results of 48 corrosion tests conducted in accordance with the CREP, using coupons protected by MIL-C-8188C. The mean corrosion rate for the specimens was 8.76 mg/hr with a standard deviation of $\pm$0.99 mg/hr. Based on these data, a truncated SSP was constructed which allowed for an early termination of testing and provided a means of determining the relative corrosion protection of an oil/inhibitor formulation in a maximum of three tests. Using the SSP in conjunction with the CREP, a maximum of three tests was required to determine whether a formulation of 2.0 w/o AI of CCI was at least as effective as, or less effective than, MIL-C-8188C.

This SSP provided an excellent means of accelerating the elimination of the less effective CCI with very reasonable levels of confidence. The acceptable quality level of this plan was 8.76 mg/hr and had an alpha risk of 0.05 associated with it. This indicates that 5% of the oil/inhibitor formulations rejected as less effective than MIL-C-8188C may have resulted, through further testing, in mean corrosion rates that were less than 8.76 mg/hr. In other words, the less effective CCI were rejected with 95% confidence. The rejectable quality level of the plan was 10 mg/hr and had a beta risk of 0.37 associated with it. This suggests that 37% of the formulations accepted may actually have had a mean corrosion rate greater than 10 mg/hr. A larger beta risk level was accepted because subsequent tests were to be performed on the most promising CCI to verify their effectiveness. The major objective of the plan was the elimination of all inferior CCI, consequently reducing the number to a maximum of 13 of the most promising.

The data generated in the corrosion tests conducted at 2.0 w/o AI of CCI are presented in Table 11, which lists the corrosion rates of metal coupons protected by a thin film[3] of MIL-L-7808H with CCI, coupons protected by a thin film of MIL-C-8188C, and the difference between these two corrosion rates for each test.

3. The oil film thickness on the coupons was approximately 13 microns (0.5 mil), as calculated according to the formula provided in Aerospace Material Specification (AMS) 3065D, 15 March 1966, Item 4.2.1.4.1.

TABLE 11

DATA FROM THE CORROSION RATE EVALUATION PROCEDURE
FOR CANDIDATE CORROSION INHIBITORS (CCI) AT 2.0 w/o ACTIVE INGREDIENT (AI)
IN MOBIL MIL-L-7808H, QRN 15F-1

Inhibitor Code Number	Corrosion Rates,* mg/hr		Difference in Corrosion Rates,* mg/hr	Status**
	MIL-C-8188C	MIL-L-7808H Plus 2.0 w/o AI-CCI		
1. PWL 80-32	8.1	0.9	-7.2	1
2. PWL 80-20	7.6	2.4	-5.2	1
3. PWL 80-28	8.3	3.6	-4.7	1
4. PWL 80-29	9.5	4.8	-4.7	1
5. PWL 80-25	9.0	4.5	-4.5	1
6. PWL 80-6	8.2	4.3	-3.9	1
7. PWL 80-22	8.3	4.8	-3.5	1
8. PWL 80-4	9.6	6.4	-3.2	1
9. PWL 80-2	8.0	5.2	-2.8	1
10. PWL 80-14	7.0	4.6	-2.4	1
11. PWL 80-7	7.8	5.5	-2.3	1
12. PWL 80-23	8.6	6.1	-2.5	1
	8.6	7.0	-1.6	
13. PWL 80-30	8.7	7.3	-1.4	1
	7.5	4.7	-2.8	
14. PWL 80-8	7.3	6.8	-0.5	2
	8.0	6.2	-1.8	
15. PWL 80-10	6.7	8.1	+1.4	2
	8.4	7.2	-1.2	
	7.8	6.1	-1.7	
16 PWL 80-38	8.7	7.9	-0.8	2
	7.6	7.5	-0.1	
	7.2	6.6	-0.6	
17. PWL 80-12	7.0	6.4	-0.6	2
	7.9	7.9	0.0	
18. PWL 80-11	7.4	7.6	+0.2	2
	7.2	6.6	-0.6	
19. PWL 80-31	7.5	8.3	+0.8	2
	9.5	8.7	-0.8	
	8.3	8.0	-0.3	
20. PWL 80-37	8.8	8.4	-0.4	2
	7.6	8.1	+0.5	
	6.8	6.8	0.0	
21. PWL 80-42	7.0	5.5	-1.5	2
	10.0	11.3	+1.3	
	8.5	9.4	+0.9	
22. PWL 80-33	8.3	8.4	+0.1	2
	8.2	8.6	+0.4	
	7.1	7.5	+0.4	
23. PWL 80-64	6.9	7.8	+0.9	2
	8.5	8.5	0.0	
	7.0	7.7	+0.7	
24. PWL 80-1	7.0	8.1	+1.1	2
	9.1	8.1	-1.0	
	7.1	9.4	+2.3	

TABLE 11 - Concluded

DATA FROM THE CORROSION RATE EVALUATION PROCEDURE
FOR CANDIDATE CORROSION INHIBITORS (CCI) AT 2.0 w/o ACTIVE INGREDIENT (AI)
IN MOBIL MIL-L-7808H, QRN 15F-1

Inhibitor Code Number	Corrosion Rates,* mg/hr		Difference in Corrosion Rates,* mg/hr	Status**
	MIL-C-8188C	MIL-L-7808H Plus 2.0 w/o AI-CCI		
25. PWL 80-39	7.1	9.0	+1.9	2
	7.7	7.4	-0.3	
	6.6	7.8	+1.2	
26. PWL 80-40	8.1	8.5	+0.4	2
	8.9	10.8	+1.9	
	7.2	8.5	+1.3	
27. PWL 80-17	6.9	9.7	+2.8	3
	8.1	10.0	+1.9	
	7.9	10.3	+2.4	
28. PWL 80-18	8.1	10.0	+1.9	3
	8.1	12.3	+4.2	
29. PWL 80-43	9.1	12.2	+3.1	3
30. PWL 80-21	8.2	11.5	+3.3	3
31. PWL 80-26	9.5	13.4	+3.9	3
32. PWL 80-35	8.6	12.8	+4.2	3
33. PWL 80-34	9.7	14.4	+4.7	3
34. PWL 80-52	8.8	15.4	+6.6	3
35. PWL 80-36	8.7	15.5	+6.8	3
36. PWL 80-62	6.7	13.8	+7.1	3
37. PWL 80-24	7.6	15.5	+7.9	3
38. PWL 80-51	10.3	18.3	+8.0	3
39. PWL 80-56	9.6	17.8	+8.2	3
40. PWL 80-53	10.6	22.0	+11.4	3
41. PWL 80-55	7.7	16.6	+8.9	3
42. PWL 80-59	6.1	15.2	+9.1	3
43. PWL 80-48	8.6	18.0	+9.4	3
44. PWL 80-44	9.3	19.0	+9.7	3
45. PWL 80-46	8.6	19.3	+10.7	3
46. PWL 80-47	8.2	19.3	+11.1	3

*All corrosion rate (CR) data represent the weight loss of 5.08 x 1.27 x 0.159 cm (2.0 x 0.5 x 0.0625 in.) AISI-1010 steel coupons, in mg/hr. The difference in CR is equal to the CR with MIL-L-7808H plus CCI, minus the CR with MIL-C-8188C. All status determinations are predicated on the Sequential Sampling Plan defined in Appendix B and the difference in CR.

**Key to status determination:

1. CCI accepted at 2.0 w/o active ingredient (AI), as being significantly more effective in corrosion protection capabilities, relative to MIL-C-8188C
2. CCI accepted at 2.0 w/o AI, as being at least as effective in corrosion protection as MIL-C-8188C
3. CCI rejected at 2.0 w/o AI based on their inferior corrosion protection capabilities, relative to MIL-C-8188C. These CCI were diluted to 0.5 w/o AI and tested with the Corrosion Rate Evaluation Procedure.

Determination of the status for the oil/inhibitor formulations involved comparing the corrosion rate of the standard metal coupon protected by 2.0 w/o AI of CCI with the acceptance and rejection limiting corrosion rates presented in Table B-1 of the Sequential Sampling Plan presented in Appendix B. For example, a metal coupon protected by 2.0 w/o AI of PWL 80-32 had a corrosion rate of 0.9 mg/hr when evaluated with the CREP, as shown in Table 11, Item No. 1. Since this value is less than the 5.8 mg/hr specified in Table B-1 for acceptance after one test (N=1), PWL 80-32 was accepted as at least as effective as MIL-C-8188C. Based on the magnitude of the negative differential corrosion rate, it is significantly more effective than MIL-C-8188C, as confirmed in subsequent tests performed in order to verify its effectiveness. On the other hand, a metal coupon protected by PWL 80-18 had a corrosion rate of 10 mg/hr in the first test, as shown in Table 11, Item No. 28. Referring back to Table B-1, since this corrosion rate was neither less than 5.8 nor greater than 10.88 mg/hr, no decision could be made after one test. A second test was completed that resulted in a corrosion rate of 12.3 mg/hr. The sum of the two corrosion rates is 22.3 mg/hr, which is greater than the 20.25 mg/hr specified for rejection after two tests (Table B-1, N=2). Consequently, PWL 80-18 was rejected as less effective than MIL-C-8188C. While a maximum of three tests was required to make a status determination, it is evident from the data that CCI which were very good or very bad required only one test.

The status of each CCI at the conclusion of testing conducted at 2.0 w/o AI appears in Table 11. Of the 46 CCI assessed at this concentration, 13 were found to be significantly more effective than MIL-C-8188C in the environment specified by the CREP. Their effectiveness is evident from the negative differential corrosion rate in each test. Another 13 CCI were found to be at least as effective as MIL-C-8188C at 2.0 w/o AI. These CCI exhibited corrosion protection essentially equivalent to that provided by MIL-C-8188C, as shown by the magnitude of the differential corrosion rates. The remaining 20 CCI were rejected on the basis of their inferior corrosion inhibiting capabilities, relative to MIL-C-8188C. It should be noted that all status determinations were predicated on the SSP with the differential corrosion rate as a means of verifying these determinations.

This same screening procedure was employed in evaluating all of the CCI that were miscible at 0.5 w/o AI in MIL-L-7808H oil. The data generated in these tests are shown in Table 12. The CCI evaluated at 0.5 w/o AI included the five that were immiscible at 2.0 w/o AI, and dilutions of the 46 that were miscible at this concentration. The latter were evaluated at 0.5 w/o AI to preclude the rejection of CCI that may have been more effective at a lower concentration. All five of the CCI that were immiscible at the higher level were found to be less effective than MIL-C-8188C in their corrosion inhibiting capabilities. Only one CCI, PWL 80-32, was found to be as effective as MIL-C-8188C at 0.5 w/o AI. The 20 CCI that were less effective at 2.0 w/o AI were also found to be less effective at 0.5 w/o AI. Therefore, on the basis of their inferior corrosion protection at the maximum and minimum concentrations, these 20 CCI were rejected from the investigation of single additive formulations. The status of each CCI

TABLE 12

DATA FROM THE CORROSION RATE EVALUATION PROCEDURE
FOR CANDIDATE CORROSION INHIBITORS (CCI) AT 0.5 w/o ACTIVE INGREDIENT (AI)
IN MOBIL MIL-L-7808H, QRN 15F-1

Inhibitor Code Number	Corrosion Rates,* mg/hr		Difference in Corrosion Rates,* mg/hr	Status**
	MIL-C-8188C	MIL-L-7808H Plus 2.0 w/o AI-CCI		
1. PWL 80-32	8.3	8.8	+0.5	1
	9.0	9.9	+0.9	
	10.1	10.2	+0.1	
2. PWL 80-20	6.3	8.9	+2.6	2
3. PWL 80-28	6.3	13.6	+7.3	2
	7.4	15.0	+7.6	
4. PWL 80-29	8.0	11.7	+3.7	2
5. PWL 80-25	7.9	12.2	+4.3	2
6. PWL 80-6	7.4	11.4	+4.0	2
7. PWL 80-22	7.9	9.1	+2.4	2
	9.5	13.1	+3.6	
8. PWL 80-4	9.5	12.6	+3.1	2
9. PWL 80-2	9.0	10.2	+1.2	2
	7.5	10.1	+2.6	
10. PWL 80-14	9.6	12.6	+3.0	2
11. PWL 80-7	7.8	12.6	+4.8	2
12. PWL 80-23	7.2	11.3	+4.1	2
13. PWL 80-30	7.8	10.8	+3.0	2
14. PWL 80-8	7.5	12.0	+4.5	2
15. PWL 80-10	8.9	12.5	+3.6	2
16. PWL 80-38	8.3	14.4	+6.1	2
17. PWL 80-12	6.5	9.0	+2.5	2
18. PWL 80-11	6.9	12.1	+5.2	2
19. PWL 80-31	7.4	12.5	+5.1	2
20. PWL 80-37	9.6	15.0	+5.4	2
21. PWL 80-42	9.5	14.7	+5.2	2
22. PWL 80-33	7.0	9.9	+2.9	2
23. PWL 80-64	9.9	15.4	+5.5	2
24. PWL 80-1	8.0	13.9	+5.9	2
25. PWL 80-39	9.8	14.7	+4.9	2
26. PWL 80-40	9.0	13.6	+4.6	2
27. PWL 80-17	6.7	13.5	+6.8	3
28. PWL 80-18	6.7	9.1	+2.4	3
	9.7	12.7	+3.0	

TABLE 12 - Concluded

DATA FROM THE CORROSION RATE EVALUATION PROCEDURE
FOR CANDIDATE CORROSION INHIBITORS (CCI) AT 0.5 w/o ACTIVE INGREDIENT (AI)
IN MOBIL MIL-L-7808H, QRN 15F-1

Inhibitor Code Number	Corrosion Rates,* mg/hr		Difference in Corrosion Rates,* mg/hr	Status**
	MIL-C-8188C	MIL-L-7808H Plus 2.0 w/o AI-CCI		
29. PWL 80-43	7.5	14.4	+6.9	3
30. PWL 80-21	6.7	15.5	+8.8	3
31. PWL 80-26	6.7	12.8	+6.1	3
32. PWL 80-35	7.6	14.8	+7.2	3
33. PWL 80-34	8.7	18.4	+9.7	3
34. PWL 80-52	7.4	16.6	+9.2	3
35. PWL 80-36	7.6	15.7	+8.1	3
36. PWL 80-62	8.3	17.9	+9.6	3
37. PWL 80-24	7.3	18.7	+11.4	3
38. PWL 80-51	8.1	17.7	+9.6	3
39. PWL 80-56	10.4	16.8	+6.4	3
40. PWL 80-53	8.0	16.9	+8.9	3
41. PWL 80-55	7.4	15.0	+7.6	3
42. PWL 80-59	6.6	15.9	+9.3	3
43. PWL 80-48	6.5	13.3	+6.8	3
44. PWL 80-44	7.9	18.2	+10.3	3
45. PWL 80-46	9.0	14.8	+5.8	3
46. PWL 80-47	7.3	16.0	+8.7	3
47. PWL 80-15	6.3	11.9	+5.6	3
48. PWL 80-16	8.1	11.6	+3.5	3
49. PWL 80-19	8.7	13.0	+4.3	3
50. PWL 80-27	7.9	12.8	+4.9	3
51. PWL 80-57	9.6	16.2	+6.6	3

*All corrosion rate (CR) data represent the weight loss of 5.08 x 1.27 x 0.159 cm (2.0 x 0.5 x 0.0625 in.) AISI-1010 steel coupons, in mg/hr. The difference in CR is equal to the CR of coupons protected by MIL-L-7808H with 0.5 w/o AI-CCI, minus the CR of coupons protected by MIL-C-8188C. All status determinations are predicated on the Sequential Sampling Plan defined in Appendix B.

**Key to Status Determination:

1. CCI accepted as being at least as effective as MIL-C-8188C at 0.5 w/o AI
2. CCI rejected as less effective than MIL-C-8188C at 0.5 w/o AI. All CCI were accepted at 2.0 w/o AI.
3. CCI rejected from the investigation of single additive formulations based on their inferior corrosion protection at 2.0 w/o and 0.5 w/o AI

at the conclusion of this phase of the investigation is indicated in Table 12.

The five CCI that were immiscible at 2.0 w/o AI and less effective at 0.5 w/o AI were subsequently evaluated at 1.0 w/o AI. Two of these, PWL 80-15 and 80-57, were immiscible at 1.0 w/o AI and were therefore rejected from this investigation. PWL 80-16 and 80-19 were miscible, but were rejected on the basis of their inferior corrosion protection at 1.0 w/o AI. PWL 80-27, however, was found to be miscible and at least as effective as MIL-C-8188C at a 1.0 w/o AI concentration. Figure 1 summarizes the screening test matrix and the results of screening 67 CCI on the basis of their solubility characteristics and corrosion protection capabilities in the environment dictated by the CREP. Table 13 is a key to the superscripts and specifies which CCI were accepted or rejected at each phase of the test series. Of the initial 67 CCI, 40 were rejected on the basis of their immiscibility or inferior corrosion inhibition relative to MIL-C-8188C.

The selection of the 13 most promising CCI was predicated on the negative differential corrosion rates in tests performed with the CREP. Triplicate tests were performed on formulations containing 2.0 w/o AI of these CCI in order to verify their effectiveness. The data generated in these tests are shown in Table 14. As shown in this table, the resulting differences in corrosion rates exhibited good repeatability for most of the formulations. Some scatter exists in the data for inhibitor PWL 80-28, but this oil/inhibitor was more effective than MIL-C-8188C in all tests. Three of the CCI (PWL 80-20, 80-29, and 80-32) exhibited excellent corrosion protection as shown by the relatively low magnitude of corrosion rates on metal coupons protected by these CCI. The data generated in these tests clearly verified preliminary determinations made on the protective capabilities of these 13 CCI.

It is interesting to note that of the 26 CCI accepted, 14 were metal sulfonates. Calcium sulfonate derivatives were very effective inhibitors in the environment of the CREP. Of the 13 most promising CCI, PWL 80-2, 80-4, 80-7, 80-14, and 80-30 are all calcium sulfonates. In a like manner, alkyl succinic acids and their derivatives (PWL 80-23, 80-25, 80-28, 80-29, and 80-32) were very tenacious inhibitors in this environment. For example, tests conducted with PWL 80-32 resulted in corrosion only on the edges of the coupons after one hour in the acidic atmosphere.

These results agree with those of Zisman (References 4 and 5) based on assessments made with the Turbine Oil Rusting Test (ASTM D-665). The effectiveness of the acids is believed to be caused by the ability of the hydrogen in the acid to coordinate with electrons on the surface of the test specimens. The tenacity of succinic acid may then be due to the fact that, being dibasic, it has twice the adsorption capacity of a monobasic acid. An interesting conclusion drawn from Zisman's work was that aliphatic alcohols are less effective on a concentration basis than the homologous primary amines and are much less effective than the homologous acids. The fundamental difference is the much shorter average lifetime of adsorption at the

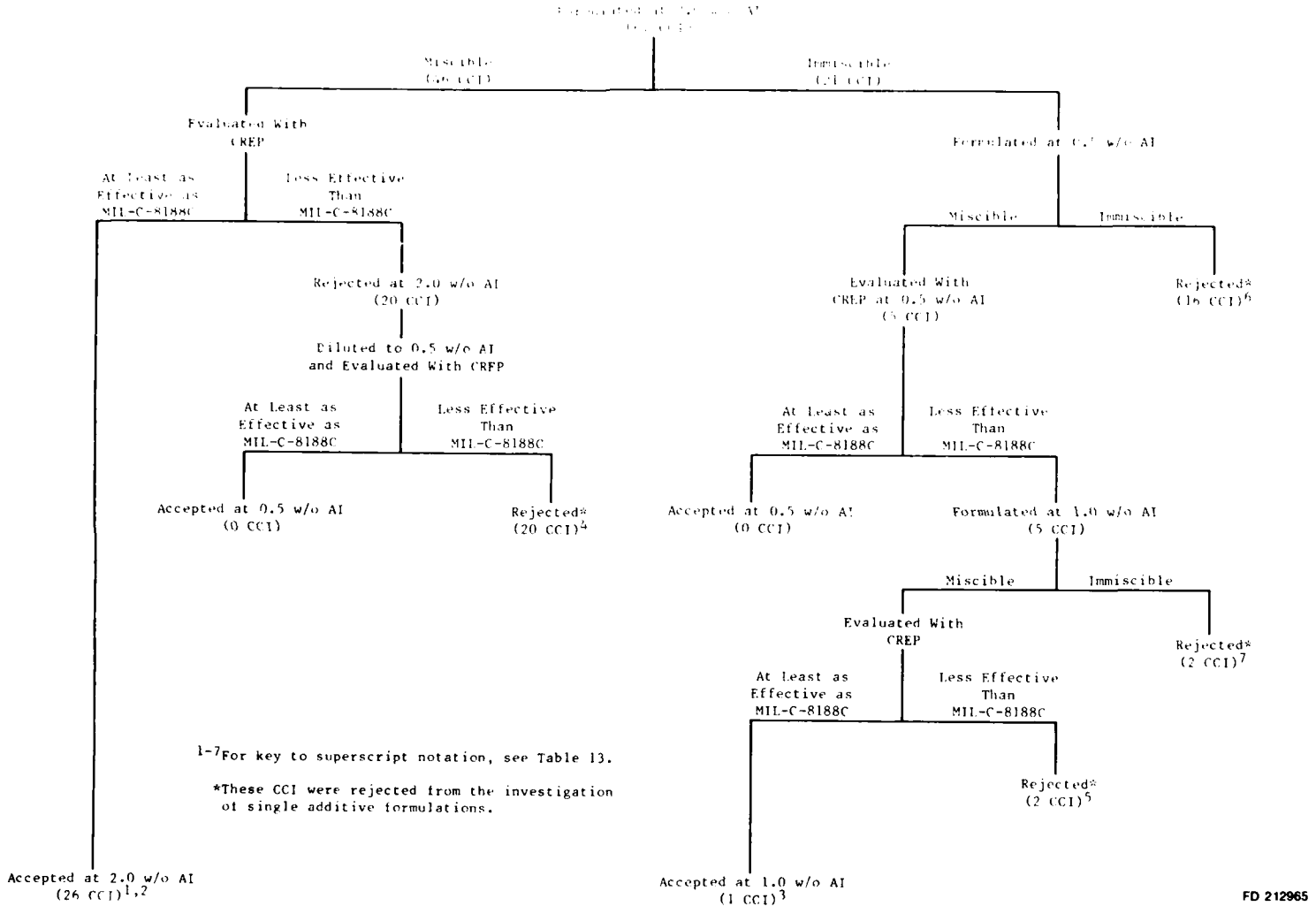


Figure 1. Miscibility/Corrosion Rate Test Results After Screening 67 Candidate Corrosion Inhibitors (CCI) With the P&WA/GPD Developed Corrosion Rate Evaluation Procedure (CREP)

TABLE 13

KEY TO THE SUPERSCRIPT NOTATION OF FIGURE 1, LISTING THE CANDIDATE CORROSION INHIBITORS (CCI) ACCEPTED OR REJECTED AT EACH PHASE OF THE SCREENING TEST SERIES

[1]CCI Accepted as More Effective Than MIL-C-8188C at 2.0 w/o AI

PWL 80-2
PWL 80-4
PWL 80-6
PWL 80-7
PWL 80-14
PWL 80-20
PWL 80-22
PWL 80-23
PWL 80-25
PWL 80-28
PWL 80-29
PWL 80-30
PWL 80-32

[2]CCI Accepted as Being at Least as Effective as MIL-C-8188C at 2.0 w/o AI

PWL 80-1
PWL 80-8
PWL 80-10
PWL 80-11
PWL 80-12
PWL 80-31
PWL 80-33
PWL 80-37
PWL 80-38
PWL 80-39
PWL 80-40
PWL 80-42
PWL 80-64

[3]CCI Accepted as Being at Least as Effective as MIL-C-8188C at 1.0 w/o AI

PWL 80-27

[4]CCI Rejected as Less Effective Than MIL-C-8188C at 2.0 w/o and 0.5 w/o AI*

PWL 80-17
PWL 80-18
PWL 80-21
PWL 80-24
PWL 80-26
PWL 80-34
PWL 80-35
PWL 80-36
PWL 80-43
PWL 80-44
PWL 80-46
PWL 80-47
PWL 80-48
PWL 80-51
PWL 80-52
PWL 80-53
PWL 80-55
PWL 80-56
PWL 80-59
PWL 80-62

[5]CCI Rejected at the Maximum Miscible Concentration of 1.0 w/o AI*

PWL 80-16
PWL 80-19

[6]CCI Rejected Due to Immiscibility at 2.0 w/o and 0.5 w/o AI*

PWL 80-3
PWL 80-5
PWL 80-9
PWL 80-13
PWL 80-41
PWL 80-45
PWL 80-49
PWL 80-50
PWL 80-54
PWL 80-58
PWL 80-60
PWL 80-61
PWL 80-63
PWL 80-65
PWL 80-66
PWL 80-67

[7]CCI Rejected at the Maximum Miscible Concentration of 0.5 w/o AI*

PWL 80-15
PWL 80-57

*These CCI were rejected from the investigation of single additive formulations.

TABLE 14

DATA FROM THE CORROSION RATE EVALUATION PROCEDURE
FOR THE MOST PROMISING CANDIDATE CORROSION INHIBITORS (CCI)
AT 2.0 w/o ACTIVE INGREDIENT (AI) IN MOBIL MIL-L-7808H, QRN 15F-1

Inhibitor Code Number	Corrosion Rates,* mg/hr		Difference in Corrosion Rates,* mg/hr
	MIL-C-8188C	MIL-L-7808H Plus 2.0 w/o AI-CCI	
1. PWL 80-32	8.1	0.9	-7.2
	9.5	0.7	-8.8
	9.8	1.0	-8.8
2. PWL 80-20	7.6	2.4	-5.2
	8.3	2.5	-5.8
	8.8	4.8	-4.0
3. PWL 80-29	9.5	4.8	-4.7
	9.2	2.6	-6.6
	9.2	5.0	-4.2
4. PWL 80-25	9.0	4.5	-4.5
	9.7	5.7	-4.0
	8.4	5.0	-3.4
5. PWL 80-6	8.2	4.3	-3.9
	9.1	5.8	-3.3
6. PWL 80-4	9.6	6.4	-3.2
	8.2	4.8	-3.4
	9.5	5.9	-3.6
7. PWL 80-2	8.0	5.2	-2.8
	9.0	5.7	-3.3
	8.7	5.2	-3.5
8. PWL 80-22	8.3	4.8	-3.5
	8.3	4.9	-3.4
	8.6	6.1	-2.5
9. PWL 80-14	7.0	4.6	-2.4
	9.0	5.5	-3.5
10. PWL 80-30	6.7	7.3	-1.4
	7.5	4.7	-2.8
	7.7	5.1	-2.6
11. PWL 80-7	7.8	5.5	-2.3
	8.5	6.2	-2.3

TABLE 14 - Concluded

DATA FROM THE CORROSION RATE EVALUATION PROCEDURE
FOR THE MOST PROMISING CANDIDATE CORROSION INHIBITORS (CCI)
AT 2.0 w/o ACTIVE INGREDIENT (AI) IN MOBIL MIL-L-7808H, QRN 15F-1

Inhibitor Code Number	Corrosion Rates,* mg/hr		Difference in Corrosion Rates,* mg/hr
	MIL-C-8188C	MIL-L-7808H Plus 2.0 w/o AI-CCI	
12. PWL 80-23	8.6	6.1	-2.5
	8.6	7.0	-1.6
	8.0	6.5	-1.5
13. PWL 80-28	8.3	7.5	-0.8
	9.4	7.8	-1.6
	8.1	6.1	-2.0
	8.3	3.6	-4.7

*All corrosion rate (CR) data represent the weight loss of 5.08 x 1.27 x 0.159 cm (2.0 x 0.5 x 0.0625 in.) AISI-1010 steel coupons, in mg/hr. The difference in CR is equal to the CR with MIL-L-7808H plus CCI, minus the CR with MIL-C-8188C protection.

oil-metal interface of the alcohol molecules as compared to the corresponding amines or acids (Reference 6).

Preliminary evaluations were performed to determine the effect of 2.0 w/o AI of CCI on the physical and chemical properties of MIL-L-7808H oil. Several of the oil/inhibitor formulations failed to conform to the MIL-L-7808H specification requirements for flash point, foaming, and/or total acid number (TAN). Since the MIL-L-7808H without CCI was in full conformance with the specification requirements, the nonconformance of the oil/inhibitor formulations was fully attributable to the CCI. Consequently, the most promising inhibitors were evaluated at lower concentrations in an effort to establish the lowest concentration of each CCI that could exhibit corrosion protection greater than that of MIL-C-8188C.

Each of the 13 most promising CCI was evaluated at 2.0, 1.5, 1.0, and 0.5 w/o AI in MIL-L-7808H using the CREP. The SSP was employed to determine the status of the CCI at each concentration. The results of these evaluations are presented in Table 15. The table also lists the status of the CCI at each AI concentration. As noted in the table, all of the CCI were found to be at least as effective as MIL-C-8188C at 2.0 and 1.5 w/o AI when evaluated on the basis of the SSP. The PWL 80-28 resulted in a differential corrosion rate of +1.0 mg/hr in two tests. The differential corrosion rate, as previously defined, indicates the corrosion rate of test coupons protected by the inhibited oil formulation and coupons protected by MIL-C-8188C in the same test kettle. The positive differential corrosion rates for PWL 80-28 at 1.5 w/o AI suggest that it may actually be slightly less effective than MIL-C-8188C. The Sequential Sampling Plan was designed to determine, on a statistical basis, if an oil formulation was less effective, or at least as effective as MIL-C-8188C. The differential corrosion rate provides a means of distinguishing between formulations which may be slightly more effective or slightly less effective than MIL-C-8188C.

The objective of the investigation of CCI at various concentrations was to determine the effective range of each inhibitor, relative to MIL-C-8188C. The lowest effective concentration of the CCI was defined as the lowest concentration of CCI that demonstrated corrosion protection superior to MIL-C-8188C. It should be noted that in these evaluations of the data presented in Table 15, determinations made on the basis of the difference in corrosion rates superseded those that were based on the SSP. Clearly, if an oil/inhibitor formulation is superior to MIL-C-8188C, it must result in negative differential corrosion rates. Evaluated on this basis, most of the CCI required a concentration of 1.5 w/o AI to provide corrosion protection superior to MIL-C-8188C in the environment specified by the CREP. Three of the 13 CCI, however, resulted in negative differential corrosion rates at 1.0 w/o AI. These were PWL 80-2, 80-4, and 80-32. In the case of PWL 80-28, a concentration of 2.0 w/o AI was required.

The aforementioned data analysis established an effective AI concentration for each of the most promising CCI relative to MIL-C-

TABLE 15

DATA FROM THE CORROSION RATE EVALUATION PROCEDURE FOR THE MOST PROMISING CANDIDATE CORROSION INHIBITORS (CCI) AT VARIOUS CONCENTRATIONS IN MOBIL MIL-L-7808H, QRN 15F-1

Inhibitor Code Number	Corrosion Rates* at 2.0 w/o AI, mg/hr			Status** (2.0 w/o AI)	Corrosion Rates* at 1.5 w/o AI, mg/hr			Status** (1.5 w/o AI)
	MIL-C-8188C	MIL-L-7808H (15F-1) With CCI	Difference in Corrosion Rates		MIL-C-8188C	MIL-L-7808H (15F-1) With CCI	Difference in Corrosion Rates	
1. PWL 80-32	8.1	0.9	-7.2	A	10.3	6.7	-3.6	A
					9.2	7.6	-1.6	
2. PWL 80-20	7.6	2.4	-5.2	A	9.4	8.1	-1.3	A
					7.8	5.5	-2.3	
3. PWL 80-29	9.5	4.8	-4.7	A	8.9	7.2	-1.7	A
					8.7	7.7	-1.0	
4. PWL 80-25	9.0	4.5	-4.5	A	9.8	8.6	-1.2	A
					9.9	8.4	-1.5	
					10.3	11.0	+0.7	
5. PWL 80-6	8.2	4.3	-3.9	A	9.7	8.0	-1.7	A
					7.6	7.1	-0.5	
6. PWL 80-4	9.6	6.4	-3.2	A	11.2	8.4	-2.8	A
	8.2	4.8	-3.4		8.6	7.6	-1.0	
					8.2	7.6	-0.6	
7. PWL 80-2	8.0	5.2	-2.8	A	9.3	6.7	-2.6	A
					7.8	7.5	-0.3	
8. PWL 80-22	8.3	4.8	-3.5	A	9.1	6.0	-3.1	A
					8.2	7.4	-0.8	
9. PWL 80-14	7.0	4.6	-2.4	A	9.7	7.2	-2.5	A
					9.2	7.5	-1.7	
10. PWL 80-30	8.7	7.3	-1.4	A	10.0	6.8	-3.2	A
	7.5	4.7	-2.8		9.5	7.9	-1.6	
11. PWL 80-7	7.8	5.5	-2.3	A	11.0	8.6	-2.4	A
					6.8	6.0	-0.8	
12. PWL 80-23	8.6	6.1	-2.5	A	9.4	6.5	-2.9	A
	8.6	7.0	-1.6		9.3	8.1	-1.2	
13. PWL 80-28	8.3	3.6	-4.7	A	9.8	10.8	+1.0	A
					7.7	8.7	+1.0	
					8.9	8.6	-0.3	

*All corrosion rate (CR) data represent the weight loss of 5.08 x 1.27 x 0.159 cm (2.0 x 0.5 x 0.0625 in.) AISI-1010 steel coupons, in mg/hr. The difference in CR is equal to the CR with MIL-L-7808H plus CCI, minus the CR with MIL-C-8188C protection.

**Status determinations of acceptance (A) are predicated on the Sequential Sampling Plan defined in Appendix B.

TABLE 15 - Concluded

DATA FROM THE CORROSION RATE EVALUATION PROCEDURE FOR THE MOST PROMISING CANDIDATE CORROSION INHIBITORS (CCI) AT VARIOUS CONCENTRATIONS IN MOBIL MIL-L-7808H, QRN 15F-1

Inhibitor Code Number	Corrosion Rates* at 1.0 w/o AI, mg/hr			Status** (1.0 w/o AI)	Corrosion Rates* at 0.5 w/o AI, mg/hr			Status** (0.5 w/o AI)
	MIL-C-8188C	MIL-L-7808H (15F-1) With CCI	Difference in Corrosion Rates		MIL-C-8188C	MIL-L-7808H (15F-1) With CCI	Difference in Corrosion Rates	
1. PWL 80-32	10.1	6.9	-3.2	A	8.3	8.8	+0.5	A
	9.7	7.9	-1.8		9.0	9.9	+0.9	
					10.1	10.2	+0.1	
2. PWL 80-20	8.6	9.7	+1.1	A	6.3	8.9	+2.6	R
	9.1	8.7	-0.4		7.9	11.7	+3.8	
	7.8	8.5	+0.7					
3. PWL 80-29	8.3	9.0	+0.7	A	8.0	11.7	+3.7	R
	11.0	10.2	-0.8					
	9.6	10.0	+0.4					
4. PWL 80-25	8.1	10.0	+1.9	A	7.9	12.2	+4.3	R
	9.9	10.1	+0.2					
	9.4	9.4	0.0					
5. PWL 80-6	10.2	9.4	-0.8	A	7.4	11.4	+4.0	R
	9.3	10.7	+1.4					
	8.7	9.1	+0.4					
6. PWL 80-4	8.8	8.7	-0.1	A	9.5	12.6	+3.1	R
	9.2	9.2	0.0					
	9.0	6.7	-2.3					
7. PWL 80-2	9.5	9.3	-0.2	A	7.5	10.1	+2.6	R
	9.1	9.4	+0.3		9.0	10.2	+1.2	
	9.7	7.7	-2.0					
8. PWL 80-22	8.7	9.5	+0.8	A	7.9	9.1	+2.4	R
	7.6	7.9	+0.3		9.5	13.1	+3.6	
	8.8	8.1	-0.7					
9. PWL 80-14	8.4	10.9	+2.5	R	9.6	12.6	+3.0	R
10. PWL 80-30	8.5	9.4	+0.9	R	7.8	10.8	+3.0	R
	9.5	12.1	+2.6		9.5	13.1	+3.6	
11. PWL 80-7	9.6	9.5	-0.1	R	7.8	12.6	+4.8	R
	9.4	12.1	+2.7					
12. PWL 80-23	9.0	10.8	+1.8	R	7.2	11.3	+4.1	R
	8.9	10.3	+1.4					
13. PWL 80-28	9.0	12.4	+3.4	R	6.3	13.6	+7.3	R
					7.4	15.0	+7.6	

*All corrosion rate (CR) data represent the weight loss of 5.08 x 1.27 x 0.159 cm (2.0 x 0.5 x 0.0625 in.) AISI-1010 steel coupons, in mg/hr. The difference in CR is equal to the CR with MIL-L-7808H plus CCI, minus the CR with MIL-C-8188C protection.

**Status determinations of acceptance (A) and rejection (R) are predicated on the Sequential Sampling Plan defined in Appendix B.

8188C. The next major task involved performing physical and chemical property characterizations of each of the oil/inhibitor formulations. The objective of this investigation was to determine which formulations failed to conform to the property requirements dictated by the MIL-L-7808H specification. The properties evaluated included foaming, flash point, total acid number, and kinematic viscosity at 99°C (210°F). Further evaluations, such as elastomer compatibility, corrosion oxidation stability, and viscosity at -54°C (-65°F), were deferred until the latter part of this program. The data generated in the chemical and physical tests are presented in Table 16. The properties of MIL-L-7808H and MIL-C-8188C are shown for comparison.

The kinematic viscosities presented in Table 16 were determined by employing the procedure detailed in ASTM D-445. These evaluations were conducted in a constant temperature viscometer bath maintained at 99°C (210°F) throughout the test. All viscometers were previously calibrated with stable Newtonian liquids with viscosities which are traceable to the NBS. The data presented in Table 16 show that the addition of the given amount of CCI resulted in a slight increase in the viscosity of the MIL-L-7808H oil. All of the formulations conformed to the specification requirements for kinematic viscosity at 99°C (210°F), which stipulates a minimum of 3.0 centistokes (cSt).

The flash points shown in Table 16 were determined by employing a Cleveland Open Cup Flash Point Apparatus and the test procedure defined in ASTM D-92. At a concentration of 2.0 w/o AI of CCI in MIL-L-7808H, four of the 13 formulations failed to meet the minimum flash point requirement of 204°C (400°F) stipulated by the MIL-L-7808H specification. The formulations that failed included those containing 2.0 w/o AI of PWL 80-20, 80-22, 80-23, and 80-28. At a reduced concentration of 1.5 w/o AI, the MIL-L-7808H/PWL 80-20 formulation exhibited an acceptable flash point of 208°C (406°F). The latter three formulations still showed flash points less than 205°C at the reduced concentration. The failure of these formulations to meet the minimum requirements may be corrected by changing CCI diluents to a higher molecular weight material. Two inhibitor manufacturers have agreed to blend the active ingredient in their CCI in MIL-L-7808H base stock for future evaluation.

The 13 most promising CCI were evaluated at two AI concentrations in MIL-L-7808H to determine their propensity to foam under the conditions of increased temperature and airflow specified by Federal Test Method (FTM) 3213. Several formulations containing 2.0 w/o AI of CCI failed to meet the MIL-L-7808H requirement for foaming. The specification allows a maximum of 100 ml of foam at any time during the 30 min test. As shown in the table, MIL-C-8188C also failed to meet this requirement as it overfoamed after three minutes. Formulations containing 1.5 w/o AI of PWL 80-7 and 80-14 overfoamed in the first five minutes of the test. Consequently, these CCI were not evaluated at 2.0 w/o AI. A formulation containing 2.0 w/o AI of PWL 80-22 overfoamed in 1.3 min.

It is interesting to note that PWL 80-2, 80-4, 80-7, and 80-14, all of which are sulfonates, produced foam volumes exceeding 100 ml at

TABLE 16

PROPERTIES OF THE MOST PROMISING CANDIDATE CORROSION INHIBITOR (CCI)/MIL-L-7808H FORMULATIONS*

CCI and Concentration of Active Ingredient	Differential Corrosion Rate,** mg/hr	Kinematic Viscosity (ASTM D445) at 99°C(210°F), cSt	Flash Point (ASTM D92), °C (°F)	Foaming Characteristics (FTM-3213), ml	Total Acid Number,*** mg KOH/g
1. MIL-L-7808H Specification Requirements	No Requirement	3.0 Minimum	204 (400) Minimum	100 Maximum	0.3 Maximum
2. Mobil Oil Co. MIL-L-7808H, (QRN 15F-1)	+6.5	3.40	216 (421)	10	0.15
3. American Oil & Supply MIL-C-8188C	-	3.41	244 (471)	Overfoamed 3.0 Min	0.11
4. PWL 80-2, 2.0 w/o	-3.2	3.70	221 (430)	370	1.44
1.0 w/o	-1.4	3.58	218 (424)	90	0.76
5. PWL 80-4, 2.0 w/o	-3.4	3.70	221 (430)	125	1.31
1.0 w/o	-0.8	3.58	210 (410)	70	0.62
6. PWL 80-6, 2.0 w/o	-3.6	-	210 (410)	75	2.23
1.5 w/o	-1.1	3.51	206 (403)	25	1.72
7. PWL 80-7, 2.0 w/o	-2.3	-	216 (421)	-	1.06
1.5 w/o	-1.6	3.57	220 (428)	Overfoamed 3.3 min	0.86
8. PWL 80-14, 2.0 w/o	-3.0	-	222 (432)	-	1.34
1.5 w/o	-2.1	3.57	214 (417)	Overfoamed 5.0 min	1.11
9. PWL 80-20, 2.0 w/o	-5.0	3.49	157 (319)	110	5.34
1.5 w/o	-1.8	3.44	208 (406)	45	3.90
10. PWL 80-22, 2.0 w/o	-3.1	3.50	154 (310)	Overfoamed 1.3 min	3.04
1.5 w/o	-2.0	3.42	180 (356)	205	2.39
11. PWL 80-23, 2.0 w/o	-1.9	3.42	149 (300)	10	4.61
1.5 w/o	-2.0	3.39	185 (365)	10	3.47
12. PWL 80-25, 2.0 w/o	-4.0	3.56	216 (420)	15	4.80
1.5 w/o	-0.7	3.47	213 (415)	15	3.58
13. PWL 80-29, 2.0 w/o	-5.2	3.56	213 (415)	15	5.04
1.5 w/o	-1.4	3.46	218 (424)	10	3.73
14. PWL 80-30, 2.0 w/o	-2.3	3.62	221 (430)	95	0.73
1.5 w/o	-2.4	3.52	218 (424)	55	0.60
15. PWL 80-32, 2.0 w/o	-8.3	3.56	207 (405)	15	7.67
1.0 w/o	-2.4	3.52	210 (410)	25	3.90
16. PWL 80-28, 2.0 w/o	-1.5	3.40	154 (310)	15	1.62

*All formulations were blended at the given concentration of active ingredient of CCI in Mobil MIL-L-7808H, QRN 15F-1.

**Corrosion Rate Evaluation Procedure (CREP)

***ASTM D664, titrate to end point of pH 11 as specified in MIL-L-7808H

1.5 and 2.0 w/o AI in MIL-L-7808H. Similarly, sulfonate-type inhibitors PWL 80-6 and 80-30 produced 75 and 95 ml of foam, respectively, when evaluated at 2.0 w/o AI. Indeed, in spite of their superior corrosion protection capabilities, sulfonates appear to have a proclivity for initiating foam generation. Only those sulfonates which exhibited superior corrosion protection were evaluated in the foaming tests. All of the sulfonates tested increased the foaming tendency of the MIL-L-7808H. Excessive foaming can be alleviated by reducing the concentration level of sulfonates, but this results in a corresponding decrease in corrosion protection capabilities. It may be necessary to reduce the propensity of these formulations to foam through addition of antifoaming additives.

The procedure used for determining the total acid number (TAN) data presented in Table 16 is defined in ASTM D-664. Formulations were titrated to an end point of pH 11.0, as required by the MIL-L-7808H specification. The Beckman SS-1 pH meter and electrodes used for these analyses were calibrated prior to each series of TAN determinations. All of the formulations failed to meet the requirements of MIL-L-7808H, which states a maximum of 0.3 mg KOH per gram of oil. This problem is currently being addressed in order to establish a method of reducing the TAN.

Table 17 shows the effect of 2.0 w/o AI of CCI on the color of the MIL-L-7808H and the extent of sedimentation during a 5 month storage period. As noted in the table, several CCI caused a significant darkening of the MIL-L-7808H at 2.0 w/o AI. Evaluation of the extent of the sedimentation involved a visual examination of formulations stored for 5 months in airtight clear glass containers at room temperature. Formulations containing PWL 80-37 and 80-38 exhibited a large amount of sediment after this storage period. Of the 13 most promising CCI, PWL 80-6 exhibited a moderate amount of sediment. PWL 80-28 and 80-30 also exhibited a small amount of sediment. The results of these evaluations indicate the importance of more controlled evaluations of the long-term storage stability of candidate formulations.

TABLE 17

THE EFFECT OF 2.0 w/o ACTIVE INGREDIENT (AI) OF CANDIDATE CORROSION INHIBITOR (CCI) ON THE COLOR OF MIL-L-7808H, QRN 15F-1, AND THE EXTENT OF SEDIMENTATION AFTER FIVE MONTHS STORAGE*

	Inhibitor Code Number	Extent of Sedimentation**	CCI Effect on Color**		Inhibitor Code Number	Extent of Sedimentation**	CCI Effect on Color**
1.	PWL 80-59	0	0	24.	PWL 80-53	0	0
2.	PWL 80-64	0	0	25.	PWL 80-47	0	0
3.	PWL 80-62	0	2	26.	PWL 80-46	0	0
4.	PWL 80-23	0	1	27.	PWL 80-48	0	0
5.	PWL 80-25	0	0	28.	PWL 80-2	0	2
6.	PWL 80-26	0	0	29.	PWL 80-4	0	2
7.	PWL 80-34	0	2	30.	PWL 80-1	0	0
8.	PWL 80-33	0	1	31.	PWL 80-22	0	2
9.	PWL 80-39	0	2	32.	PWL 80-7	0	1
10.	PWL 80-40	0	2	33.	PWL 80-8	0	2
11.	PWL 80-29	0	0	34.	PWL 80-12	0	1
12.	PWL 80-32	0	0	35.	PWL 80-14	0	1
13.	PWL 80-36	0	2	36.	PWL 80-35	0	0
14.	PWL 80-42	0	0	37.	PWL 80-24	1	1
15.	PWL 80-43	0	0	38.	PWL 80-28	1	0
16.	PWL 80-44	0	0	39.	PWL 80-30	1	1
17.	PWL 80-21	0	0	40.	PWL 80-17	1	1
18.	PWL 80-18	0	0	41.	PWL 80-10	1	1
19.	PWL 80-20	0	0	42.	PWL 80-11	1	2
20.	PWL 80-52	0	0	43.	PWL 80-31	2	1
21.	PWL 80-55	0	0	44.	PWL 80-6	2	2
22.	PWL 80-56	0	0	45.	PWL 80-37	3	2
23.	PWL 80-51	0	0	46.	PWL 80-38	3	2

*All formulations consisted of 2.0 w/o AI of CCI in MIL-L-7808H. Formulations were stored at room temperature, 25°C (77°F).

**Key to sediment and color codes:

Sediment Code		Color Code	
0	No visible sediment	0	No change in color of MIL-L-7808H
1	Small amount of sediment	1	Slightly darkened
2	Moderate amount of sediment	2	Significantly darkened
3	Large amount of sediment		

SECTION V

CONCLUSIONS

Although the P&WA/GPD-developed Corrosion Rate Evaluation Procedure (CREP) is an expedient preliminary screening test, it currently lacks the precision and repeatability desired for the final selection of a corrosion inhibiting engine oil for the ALCM F107 engine. It appears that the atmosphere of the current CREP may be too aggressive to obtain the desired repeatability. Future investigations conducted in this program should include evaluations of the various parameters of the CREP in order to determine their effect on the magnitude and precision of the resulting corrosion rate determinations. Suggested investigations are included in Section VI, Recommendations.

Based on the results of corrosion tests conducted in this program, it appears that the sulfonates and organic acid derivatives are the most promising candidate corrosion inhibitors for the ALCM F107 engine lubricating oil. However, in spite of the excellent corrosion protection afforded by these inhibitors, there is a concomitant increase in the TAN and/or foaming tendencies of oil formulations containing these compounds. These problems may be alleviated through a modification of the inhibitor or through additional formulation.

It has been demonstrated in this phase of the program that an active ingredient concentration of 1.5 to 2.0 w/o is necessary to provide the required corrosion protection. At this concentration, it is imperative that the diluent used in the CCI be a material that will not have a deleterious effect on the operational characteristics of the MIL-L-7808H oil. Inhibitors in low molecular weight solvents were shown to decrease the flash point of the oil/inhibitor formulation below the acceptable minimum requirement of the MIL-L-7808H specification.

SECTION VI

RECOMMENDATIONS

Prior to further evaluations of candidate corrosion inhibitors, an investigation should be performed to improve the repeatability and precision of the CREP with a concurrent reduction in the severity of the test. This investigation should involve evaluations of the various test parameters of the CREP and their effect on the resulting corrosion rate determinations.

One of the most important variables of any corrosion test is the pretest and post-test treatments of the metal test coupons. During this investigation, various methods of surface preparation should be evaluated. The alternative methods described in ASTM D-1748 should be assessed in addition to the current method specified by the CREP. Each surface should be evaluated on the basis of the repeatability of surface finish as determined by profilometer. Scanning electron microscopy (SEM) should be employed to determine the extent of grit embedment in metal surfaces prepared by each method. This investigation should also include the development of a more standardized post-test cleaning procedure to remove oxides generated during the corrosion test without attacking the base metal of the coupon.

An increase in precision and repeatability may be established by varying the test coupon dimensions. Increasing the surface area of the coupons may result in a substantial decrease in the data variations observed in the current CREP.

Other important variables that may affect the severity of the test include the humidifying solution, temperature, airflow rate, and residence time. An optimum set of test parameters should be established through investigations to determine the corrosion rate as a function of each test variable. The precision and repeatability of the modified CREP should be determined and followed by the development of a revised Sequential Sampling Plan.

In order to verify the selections of the more promising candidate corrosion inhibitors established in the first part of this program, representative samples of each class of inhibiting compound should be evaluated with the modified procedure. Subsequent evaluations should emphasize derivatives and variations of the more promising compounds.

Due to the variations in composition of the different base stocks of MIL-L-7808H turbine engine lubricant, the most promising CCI should be evaluated in base stocks with various Qualified Products List (QPL) designations. All preliminary tests should employ MIL-L-7808H with the QPL designation 15F-1, with subsequent evaluations of the most promising CCI in base stocks having designations 11E-1 and 15E-1.

A correlation between the results of the CREP and the corrosion as found under actual engine storage conditions has yet to be examined. In order to verify the CREP with regard to the "in situ" corrosion

rate of the Air-Launched Cruise Missile (ALCM) engine components, a series of panels should be exposed to a representative storage environment for a prescribed period of time. This evaluation would provide a means of correlating the corrosion rates observed in actual field tests with those of the CREP.

APPENDIX A

CORROSION RATE EVALUATION PROCEDURE (CREP) FOR THE GRAVIMETRIC DETERMINATION OF CORROSION RATES OF ENGINE HARDWARE WITH JET ENGINE LUBRICANTS

1. SCOPE

The following Corrosion Rate Evaluation Procedure (CREP) covers the determination of the relative protection afforded by thin film oil coatings against corrosive attack on engine components that are subjected to long periods of inactive storage.

2. SUMMARY OF METHOD

A vapor blasted, precleaned, tared metal strip, conforming to the AISI 1010 specification requirements and coated with the test oil, is suspended for 60 min in the 99 $\pm$ 1°C vapor phase of a boiling acetate buffer solution. At the end of this exposure cycle, the strip is cleaned, dried, and weighed to $\pm$0.1 mg to calculate metal loss from corrosive attack.

3. SIGNIFICANCE

This method is used to determine the comparative degree of corrosion protection furnished by different oils as evaluated by this procedure.

4. APPARATUS, MATERIALS, AND REAGENTS

4.1 Reaction kettle[1] (Kimax™ 33700), 1000 ml capacity, complete with cover having finely ground flange for tight seal. Each cover has four standard taper (T) 24/40 female joints.

4.2 Graham condenser,[2] water cooled, 400 mm jacket length, with a $\overline{T}$ 24/40 male joint to match one of the four female joints of the kettle cover.

4.3 Hot plate,[3] electric, Thermolyne Model SP-13115 or equivalent, suitable for running two tests simultaneously.

4.4 Mass flowmeter,[4] Models LF-100 and LF-1K have been found to be satisfactory.

4.5 Thermometers,[5] mercury filled (-10 to 360°C) or ASTM 40C (72 to 126°C) have been found to be satisfactory.

4.6 Analytical balance, readability 0.1 mg; Sartorius, Model 2842SR, or equivalent has been found to be satisfactory.

4.7 Desiccator, Fisher Scientific Company catalog No. 8-632, or equivalent.

4.8 Micro-Wipes.[6]

4.9 IBM typewriter brush.[7]

4.10 Rubber Stoppers, No. 5, Buna-N compound. (Neoprene composition is not acceptable due to reactivity with vapors from boiling acetate buffer solution.)

4.11 Novaculite 200,[8] 200-grit powder wet abrasive compound used for vapor-blasting AISI 1010 test specimens.

4.12 Toluene,[9] purified, for cleaning test specimens.

4.13 Acetone,[10] reagent grade, for cleaning test specimens.

4.14 Buffer solution,[11] pH 4.63, heated solution used as matrix for corrosive vapor environment.

4.15 Compressed dry air, size A cylinder, complete with two-stage regulator.

4.16 Metering valve for airflow control, Whitney Model 22RS4, or equivalent.

4.17 Air injection tube, boro-silicate glass tubing, 23.0 cm in length, 0.3 cm ID with outlet end flared to 0.6 cm ID. The larger ID flared tube end eliminates air blockage from vapor condensation.

4.18 Boiling beads, Kimax, (Kimble 13500), 3 mm dia.

4.19 Universal timer,[12] Model 170, Dimco Gray Company, Dayton OH, or equivalent.

4.20 Suspension wire, AMS-5837 Inconel, 21-gauge, cut and formed to required geometrical configuration.

4.21 Dow Corning stopcock grease, or equivalent (silicone grease).

4.22 Alconox detergent, Alconox Inc., VWR Scientific Inc., PO Box 13007, Sta. K, Atlanta GA.

Acceptable Sources for Apparatus, Materials, and Reagents:

1. Fisher Scientific Company, PO Box 829, Norcross GA, catalog No. 11-847B
2. Ace Glass Company, Vineland NJ, catalog No. 5953-15
3. VWR Scientific Inc., PO Box 13007, Sta. K, Atlanta GA, catalog No. 33922-950
4. Matheson Gas Products, East Rutherford NJ

5. Ace Glass Company, Vineland NJ, catalog No. 8315-34, or Fisher Scientific Company, PO Box 829, Norcross GA, catalog No. 13-487-10D
6. Scott Paper Company, Philadelphia PA, Scottbrand 05310
7. International Business Machines Company
8. Malvern Minerals Company, Inc., PO Box 1246, Hot Springs National Park AR
9. Fisher Scientific Company, PO Box 829, Norcross GA, catalog No. T-323
10. Fisher Scientific Company, PO Box 829, Norcross GA, catalog No. A-17-S
11. Fisher Scientific Company, PO Box 829, Norcross GA, catalog No. So-B-100
12. VWR Scientific Inc., PO Box 13007, Sta. K, Atlanta GA, catalog No. 26396-508

5. TEST SPECIMEN PREPARATION

Sample strips of material conforming to AISI 1010 specification requirements with dimensions 1.27 x 5.08 x 0.16 cm (0.5 x 2.0 x 0.06 in.) are used as corrosion test specimens. A hole, 0.24 cm (0.09 in.) in dia and centrally located 0.32 cm (0.12 in.) from the longitudinal end of the specimen, is used for suspension during the oil immersion and heated test cycles. Specimen identification integrity is maintained by the use of die-cut 0.32 cm (0.12 in.) high numbers positioned immediately below the suspension hole.

All surfaces and edges of the specimen are vapor blasted for 45 sec, using 200 grit novaculite as the abrasive medium. The vapor blast nozzle operates at a pressure of 0.62 MN/m^2 (90 psi), and is held 15 to 20 cm (6 to 8 in.) from the specimen during the surface preparation. After vapor blasting, the specimens are rinsed with water and cleaned by wiping with toluene-wetted Micro-Wipes to remove any remaining residue. The specimens are then immersed in boiling toluene, removed, and immediately immersed in boiling acetone. Upon removal from the acetone, the specimens are flash dried and placed in a desiccator while cooling to ambient temperature; 30 min is sufficient time for cooling. The prepared specimens are weighed to $\pm$0.1 mg prior to testing. Specimen cleanliness is maintained by handling with forceps and wearing cotton gloves.

6. REACTION KETTLE PREPARATION

Cleaning of the reaction kettle prior to test cycles is accomplished by first wiping both flanges of the assembly with Micro-Wipes to remove residual silicone grease. The kettle and cover are cleaned by washing with hot water and Alconox detergent, followed by rinsing with deionized water. The assembly is then rinsed with acetone and dried by blowing with dry compressed air. The condensers are flushed with deionized water prior to each test cycle.

7. CORROSION TEST APPARATUS

The dual apparatus, shown in Figure A-1, is the system being used for P&WA/GPD evaluation of the corrosion resistant properties of lubricants and lubricant/inhibitor formulations. The two 1000 ml reaction kettles placed on the dual hot plate are equipped with water-cooled condensers to reflux the ascending acidic vapors from the kettles.

The four female ℸ 24/40 joints on the reaction kettle cover are utilized as follows: one for the water-cooled condenser, one for the thermometer and air injection tube assembly, and the remaining two for No. 5 rubber stoppers with attached specimen suspension wires. The thermometer is positioned to ensure that the top of the mercury reservoir bulb is 13 cm (5.1 in.) above the base of the kettle. The air injection tube is positioned in such a manner that the enlarged outlet end is 9 cm (3.5 in.) above the base of the kettle. The specimen suspension wires are formed in such a manner that the bottom edge of the specimen is 11 cm (4.3 in.) above the base of the kettle.

8. INITIAL TEST SETUP

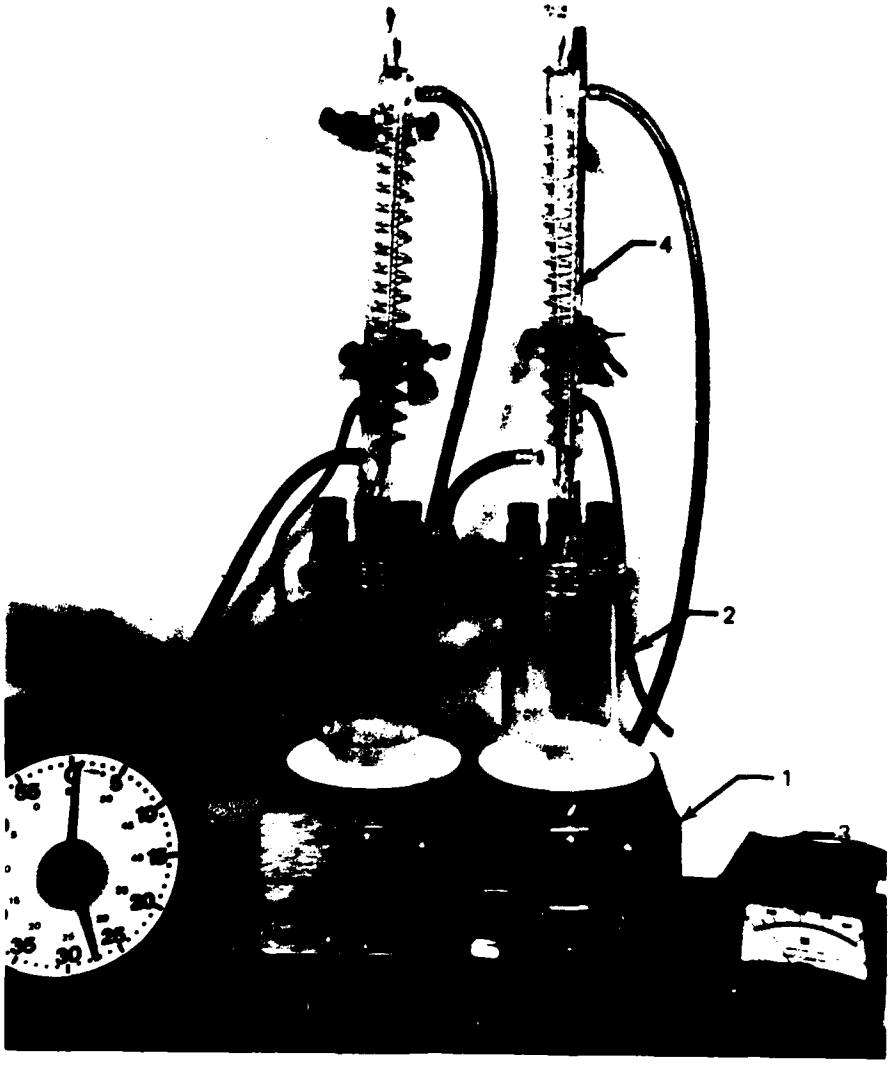
After the aforementioned corrosion test apparatus is assembled, a 100 ml aliquot of buffer solution is added to each reaction kettle. Boiling beads are added to the solution to eliminate splatter of solution on the specimens during the heating cycle. The ground glass flange of the reaction kettle is coated with a thin film of silicone grease to prevent condensate leakage past the mating flanges of the kettle and the cover.

A regulated flow of clean dry air is provided from a compressed air cylinder with the air flowrate being controlled by a micrometer-type valve and measured with a mass flowmeter. An air flowrate of 45 cc/min to each reaction kettle is established before the buffer solution starts boiling to preclude condensate blockage of the air injection tube.

9. CORROSION TEST PROCEDURE

The test specimens, previously vapor blasted, cleaned, and weighed to $\pm$0.1 mg, are immersed in the test oil or oil/inhibitor formulation for 5 min. At the end of this immersion cycle, the specimens are removed and suspended in a vertical position for a draining period of 15 min. During this period of time, the required 99 $\pm$ 1°C vapor phase temperature and the 45 cc/min air flowrate are checked to verify temperature and air flow equilibrium. At the end of the 15 min draining cycle, excess oil at the bottom edge of the test specimens is removed by blotting with a Micro-Wipe.

The test specimens, suspended by the suspension wires attached to No. 5 stoppers, are placed into the two remaining ℸ 24/40 female joints in the reaction kettle for a test period of 60 min. Figure A-2 shows a closeup view of a reaction kettle with specimen strips, thermometer, and air injection tube.

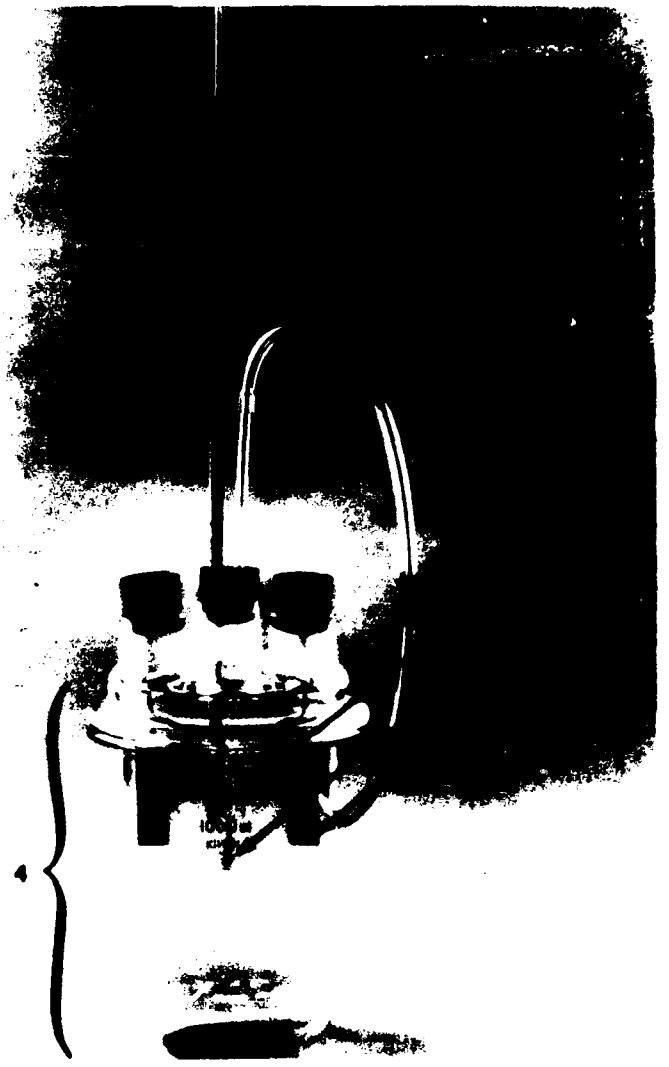


FE 187373

1. Thermolyne Hot Plate
2. Reaction Kettle
3. Mass Flowmeter
4. Water Cooled Condenser
5. Laboratory Timer and Alarm

FD 189818

Figure A-1. Dual Reaction Chamber With Peripheral Equipment



FE 187374

1. Test Specimen
2. Air Inlet Tube
3. Thermometer
4. Reaction Kettle

FD 189819

Figure A-2. Reaction Chamber With Sample Strips

The specimens are removed from the reaction kettles at the end of the 60 min test cycle. The adhering oxides and oil are removed from the specimens by wetting with acetone and brushing with an IBM typewriter brush. Secondary cleaning of the test specimens is accomplished by wiping all surfaces and edges with Micro-Wipes saturated with acetone. This is followed by wiping all surfaces and edges with Micro-Wipes saturated with toluene until no further oxides can be removed. Final cleaning of test specimens is done by immersion of each specimen in boiling toluene, followed by immersion in boiling acetone. The specimens are then flash dried and placed in a desiccator to cool.

After 30 min, the specimens are weighed to ± 0.1 mg, and the weight differential from pretest weighing is calculated.

10. SUPPLEMENTARY NOTES CONCERNING THE TEST PROCEDURE FOR THE GRAVIMETRIC DETERMINATION OF CORROSION RATES INVOLVING MIL-L-7808H TYPE AIRCRAFT LUBRICATING OILS

It has been found that certain variations in apparatus or test conditions will affect the test duration and/or results. These have been summarized as follows:

a. Less than 100 cc of acetate buffer solution will require a longer time to attain vapor phase temperature equilibrium. Normal temperature equilibration time using 100 cc of liquid is 30-45 min after liquid boiling begins. When a larger volume of acetate buffer is used, a longer equilibration time is required. No test benefits are derived from using a larger volume of liquid.

b. Some experimental work has been done at lower and higher air flowrates with the other parameters held constant. Lower air flowrates tend to lower the rate of corrosive attack, while higher air flowrates are prone to produce erratic test data. As a result of this experimental work, the air flowrate must be carefully controlled to ensure test validity.

c. The position of the air injection tube is very important in that the outlet end of the air tube must be below the thermometer with air flow started before applying heat to the reaction kettle. This early air flow prevents condensation within the air tube, thus precluding splatter of the condensate with initiation of air flow.

d. Test specimens should be used within one hour of vapor blast treatment to prevent possible oxidation resulting from long residence periods before testing.

e. Extreme care should be used during the post-test cleaning of specimens to remove all possible oil and corrosion residue prior to immersion in the boiling toluene and acetone.

APPENDIX B

A TRUNCATED SEQUENTIAL SAMPLING PLAN FOR OIL ADDITIVE MIL-C-8188C COMPARISON STUDY

1. INTRODUCTION

A sequential sampling plan has been developed for the analytical investigations being conducted at the P&WA/GPD Advanced Fuels and Lubricant Group involving MIL-L-7808H oil blended with candidate corrosion inhibitors. This appendix offers a truncated sequential sampling plan requiring as little as one with no more than three corrosion rate determinations (N) required for an acceptable risk level (Alpha) of 5%.

2. RECOMMENDATIONS

Table B-1 presents the truncated sequential sampling plan with an acceptable risk level (Alpha) of 0.05 and a rejectable risk level (Beta) of 0.37. This sampling plan will result in a decision within a maximum of three tests.

TABLE B-1

SEQUENTIAL SAMPLING PLAN,
WITH AN ALPHA OF 0.05 AND A BETA OF 0.37

N	Accept (<)		Reject (>)	
	mg/hr	Average	mg/hr	Average
1	5.8	5.8	10.88	10.88
2	15.21	7.6	20.25	10.13
3	29.63	9.88	29.6301	9.88

Note: The Acceptable Quality Level (AQL) is 8.76 mg/hr and the Rejectable Quality Level (RQL) is 10.0 mg/hr.

3. DISCUSSION

Sequential sampling allows early termination of testing when the material being tested is very good or very bad. For example, if the test material had a corrosion rate greater than 10.0 mg/hr or less than 5.0 mg/hr, testing could be terminated with an average of only 1.25 tests.

The Beta risk is higher in the truncated case than in the original sequential sampling plan. This means that material having a corrosion rate higher than 10.0 mg/hr has a greater probability of being accepted. The Alpha risk remains the same.

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